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Gasification of Dutch sewage sludge in supercritical water

commissioned by STOWA / NL

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Final Report

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1 Introduction

In the Netherlands every person is responsible for the production of 190 g sewage sludge per day which accumulates in municipal as well as in industrial sewage purification plants. The sewage sludge delivered from STOWA originates from the cities Lelystad and Oijen. Figure 1 shows the flow diagram of the sewage purification plant of Oijen.

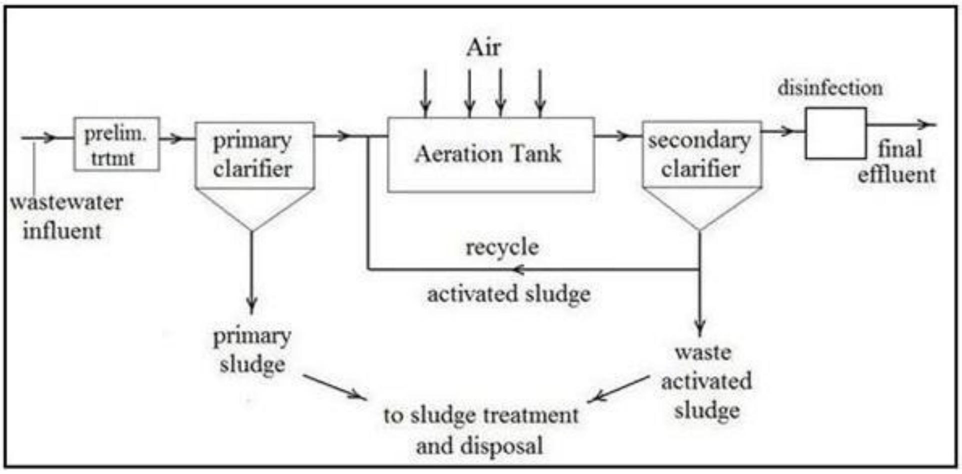


Figure 1: Activated sludge waste water treatment flow diagram.

First in the preliminary treatment the wastewater is released from large particles and the main part of sand. After this step the wastewater passes the primary clarifier, aeration tank, secondary clarifier to finally get discharged into the environment. During this process primary sludge and waste activated sludge accrue. Primary sludge contains large particles, as faeces, fruit residues, toilet paper and corks while the consistence of waste activated sludge is more homogeneous. The primary sludge and some part of the waste activated sludge are mixed in a ratio of about 1:1 and the resulting sludge is concentrated to the desired dry matter content by a centrifuge.

The main difference between the process of Oijen to that of Lelystad (tested only in the Lab scale) is that the latter one has no primary clarifier and during the biological treatment in the aeration tank no aluminium chloride ($AlCl_3$) is added for the phosphate precipitation (source STOWA).

The composition of sewage sludge is strongly dependent from the ingredients of the wastewater, the process method of the sewage purification plant and the additives used in the appropriate facility. The raw sludge consists of various chemical species in the neutral pH-range with a typical dry matter content of 3,5 wt.%. Due to its high portion of organic compounds sewage sludge counts as wet residual biomass respectively organic waste. However heavy metals like zinc, copper, lead, cadmium, nickel, quicksilver and chrome and also pharmaceutical residues of the wastewater accumulate in the sewage sludge.

One possibility to utilize the waste product sewage sludge is the hydrothermal gasification, which takes place in supercritical water. The main products are H_2, CO_2, CO and CH_4. However the reaction conditions, especially the temperature, affect the gasification products of the biomass. The hydrothermal gasification is the most energetic efficient chemical conversion of wet biomass with a water content higher than 40 wt.%. The major advantages of the use of supercritical water as reaction medium are: the density of supercritical water at 600 °C and 250 bar is 87 kg/m^3, much higher than for conventional gasification. This leads to a much higher space-time yield. Increased thermal conductivity and higher temperature will promote the endothermic reforming reaction. The required compression work is low due to the low compressibility of the liquid fuel. The gas product on the other side is compressed to about 25 MPa, which is optimum for short time storage.

1.1 Gasification of sewage sludge in supercritical water

Several publications report on lab-scale experiments on the gasification of sewage sludge in supercritical water. Most of them report experiments performed in batch autoclaves. These results can not be directly compared with a continuous flow set-up associated with much shorter residence times.

Acelas et al. carried out SCWG experiments in a batch autoclave reactor with a reaction volume of 45 mL at different temperatures from 400 to 600 °C. Also the residence time was varied to investigate it´s influence on the gasification efficiency. In a typical experiment sewage sludge with a dry matter content of 3,3 wt% was used. By increasing the temperature they obtained higher gasification efficiencies (52 % at 600 °C for residence times of 15 min and more).

Gong et al. investigated the influence of the composition of the sewage sludge on the gasification in SCW. The water content ranged between 73 and 89 wt% the organic matter content of the dry sewage sludge varied from 29 to 73 wt%. The experiments were carried out in a high pressure autoclave with an internal volume of 100 mL at almost constant operating conditions (400 °C, 60 min, 23 MPa). In a typical run sewage sludge with a water content of 73.48 to 88.51 wt% was used. The results of the SCWG showed that the gas production increased linearly with the increasing organic matter content in the dewatered sewage sludge. They supposed that the differences in hydrogen content of the product gas arose from differences in the composition of inorganics, water content and pH value of the dewatered sewage sludge samples. The formation of char and coke was increasingly observed at low water contents.

Also Chen et al. carried out experiments in a batch reactor (reactor chamber: 140 mL) to investigate the gasification of sewage sludge in near and super-critical water. For a typical run sewage sludge with a dry matter content of about 9 wt% was used and the pressure ranged from 21 to 25 MPa. The temperature was varied from 350 to 450 °C, while residence times of 5 to 25 minutes were adjusted. The aim of the experiments was to investigate the effect of temperature on the development of the reaction process and the production of gases. Therefore they analyzed the characteristics of solid and liquid products in detail. The results indicated that organic contents in sewage sludge are almost completely dissolved and hydrolyzed in water at 425 °C.

1.2 Short description of the project

STOWA ordered 15 lab scale experiments and two pilot scale experiments to IKFT / KIT.
Detailed analytical work should enable a well-founded evaluation of the experiments and generate data valuable for planning a commercial plant for the gasification of sewage sludge in supercritical water.
STOWA charged a steering committee with the control of the scientific technical progress and route of the experimental program. Leon Korving was charged with the coordination of the whole project.

1.3 Targets of the project

The objectives for the lab scale tests were:

- to collect preliminary information on sludge conversion in supercritical water, especially with regard to the degree of conversion that is feasible, the gas production and the behavior of N- compounds and other minerals. With regard to the N-compounds it is important to confirm that ammonia will be the prime N-containing product.
- to study the basic process parameters like temperature, pressure, concentration of feed and mean residence time.

For KIT these tests were important to gain insight in the expected behavior of the sludge in its VERENA pilot plant.

The objectives for the pilot-scale tests were:

- to increase the general knowledge on supercritical water gasification of sewage sludge.
- to result in an assessment of the feasibility of supercritical water gasification of sewage sludge.
- Sewage sludge is the most important wet waste biomass. Thus the tests will act as a stimulant for the general development of supercritical water gasification.

1.4 Description of the process

The VERENA facility (German acronym for “experimental facility for the energetic exploitation of agricultural matter”) is a dedicated plant for the biomass gasification in supercritical water. The pilot plant VERENA (see Figure 2) has a total throughput of 100 kg wet feed per hour and is designed for an operating pressure of up to 35 MPa and a maximum reactor temperature of 700 °C. A measured quantity of the feed is transferred into the stirred preparation tank (volume 1,5 m^3) with a connected mill. For the pilot scale test with sewage sludge the stirred preparation tank was not used. Instead an IBC served as storage tank from which the feed was pumped directly into the plant. The feed is dosed by a mass flow controlled high pressure pump to the reaction system. The feed flow passes a double-pipe heat exchanger and is heated in counter current flow by the hot effluent from the reactor. Subsequent the preheated feed flow is lead into the salt separation system where it is indirectly electrically heated. A cyclone is attached to the lowest part of the salt separation system to remove the brines. After this step the feed passes the reactor, which is heated by externally generated flue gas. It has a total volume of 30 L, can be operated up to 700 °C and is divided into two parts. The first part, the preheater, is a tubular reactor which acts primarily as heat exchanger; the second part is a slim vessel. Under supercritical conditions, the gaseous reaction products are soluble in water and leave the reactor as a homogeneous phase, pass the heat exchanger and a cooler. By cooling down the product gases separate from the water phase. After phase separation, the pressure is reduced in two steps. The second separator is equipped with an integrated CO_2 scrubber. This option was not used for the pilot scale tests for STOWA, since the direct utilization of the product gas in an internal combustion motor is possible and more economic. The effluent is led to a store tank and the salt concentrates from the cyclone and the sump of the reactor are each led into a tank on a balance. The VERENA facility is controlled by a process control system on base of the application software WINCC.

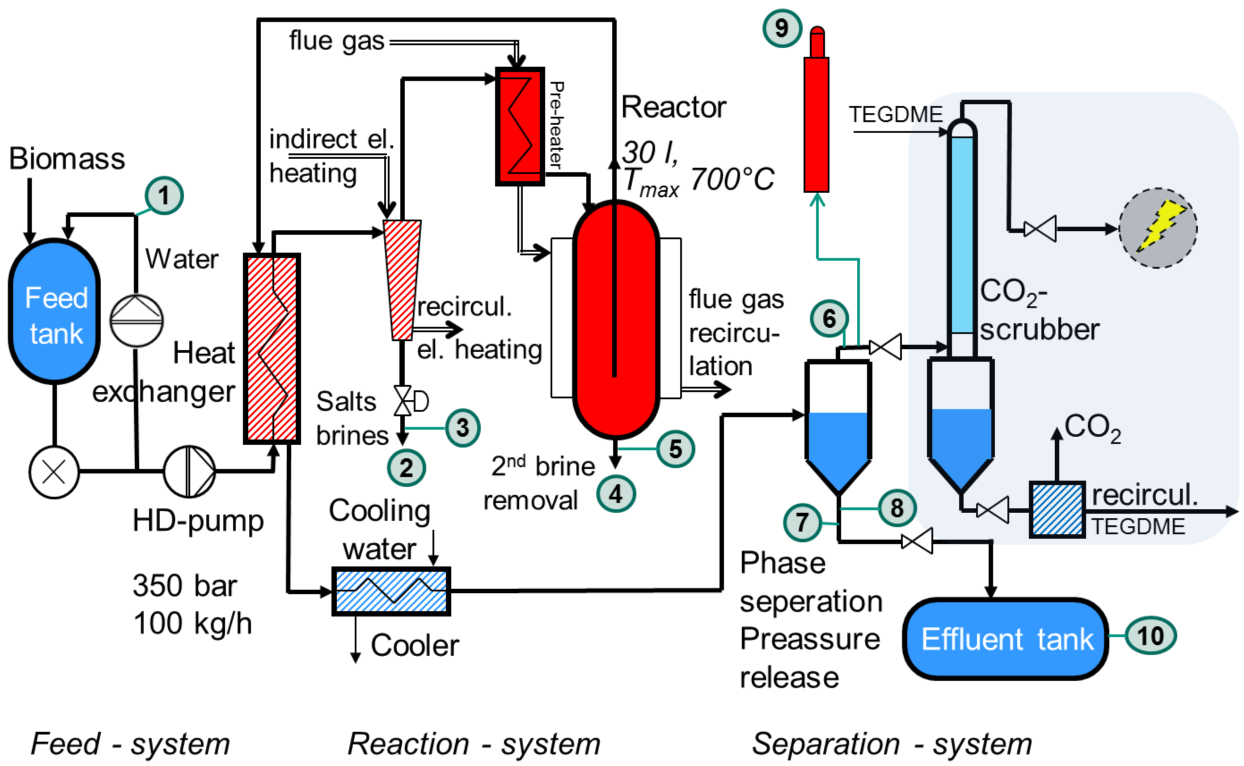


Figure 2: Pilot plant VERENA – flow sheet.

Samplings points:

1. feed sample
2. salt concentrate - cyclone (consisting of solid and aqueous phases)
3. lean gas cyclone
4. second salt concentrate - reactor
5. lean gas sump reactor
6. gaseous phase
7. aqueous phase
8. lean gas separator
9. high pressure sample
10. waste water

The amount of the lean gas streams (3, 5 and 8) as well as the amount of the product gas (6) is measured with gas meters.

2 Lab scale experiments

The Lab-scale tests were performed in a dedicated continuous flow test rig. Throughput is up to 1000 g wet feed / h. The test rig is equipped with a piston –like feeding system capable to feed even viscous feeds (only requirement is not to plug a tube with 8 mm i.d.). The reactor is a tube with 18 mm i.d. Temperatures up to 700 °C and 300 bar pressure can be realized in this reactor. The salt separation system is working satisfactory. Experimental times of several hours (up to 3,4 kg feed at once) are regularly performed. Usually the feed is treated with a mixer and some potassium salts are added as catalytically active components.

2.1 Experimental set up

The LENA test rig (German acronym for "laboratory plant for the energetic exploitation of agricultural matter") is the apparatus that is used for the supercritical gasification of sewage sludge. Figure 3 shows its flow sheet. The main components are a cylindrical tank filled with the sewage sludge, a down-flow reactor with preheater and a separate brine (salt) separation system.

The sewage sludge is separated spatially from the hydraulic system of the LENA test rig by a piston. During the experiments a HPLC pump (P04) is responsible for the transportation of the sewage sludge into the preheater with flow rates of 1 – 3 mL/min. The same function is fulfilled by the piston pump P01, which is used if flow rates higher than 3 mL/min are required. The preheater made of Inconel 625 consists of two heaters (HZ 1 and HZ 2). A tee after the preheater separates the inorganic solid salts from the feed by inertial separation. The inorganic salts get into the brine separation system, which is partially heated with heater HZ 9 directly after the preheater. The feed reaches the reactor, which is also made of Inconel 625. Six heaters (HZ 3 – HZ 8) heat the reactor.
The inorganic salts leave the preheater by successively opening and closing the valves V36 and V37 at certain time intervals. A candle filter (F02) as strainer is connected upstream of these valves. After passing the tee the inorganic salts partially dissolve again in subcritical water. The weight of the salt concentrate is registered by a balance (Salt).

After the hydrothermal gasification the effluent and the product gas leave the reactor at its bottom to pass the candle filter F01. This filter is connected upstream of a pneumatically controlled back-pressure regulator (V34). V34 regulates the pressure of the test rig. However the candle filter F01 can be bypassed. In this case the pneumatically controlled back-pressure regulator V35 controls the pressure of the apparatus. A scale (Outlet) displays the weight of the effluent. The amount of product gas is measured with a gas meter, which is connected downstream of the balance Outlet.

For the sampling of the liquid and gaseous phases serve round bottom flasks. Each of these round bottom flasks possesses an outlet for the liquid phase and a septum for the gas sampling with a gas syringe.

The whole LENA test rig is controlled by a process control system on base of the application software WINCC.

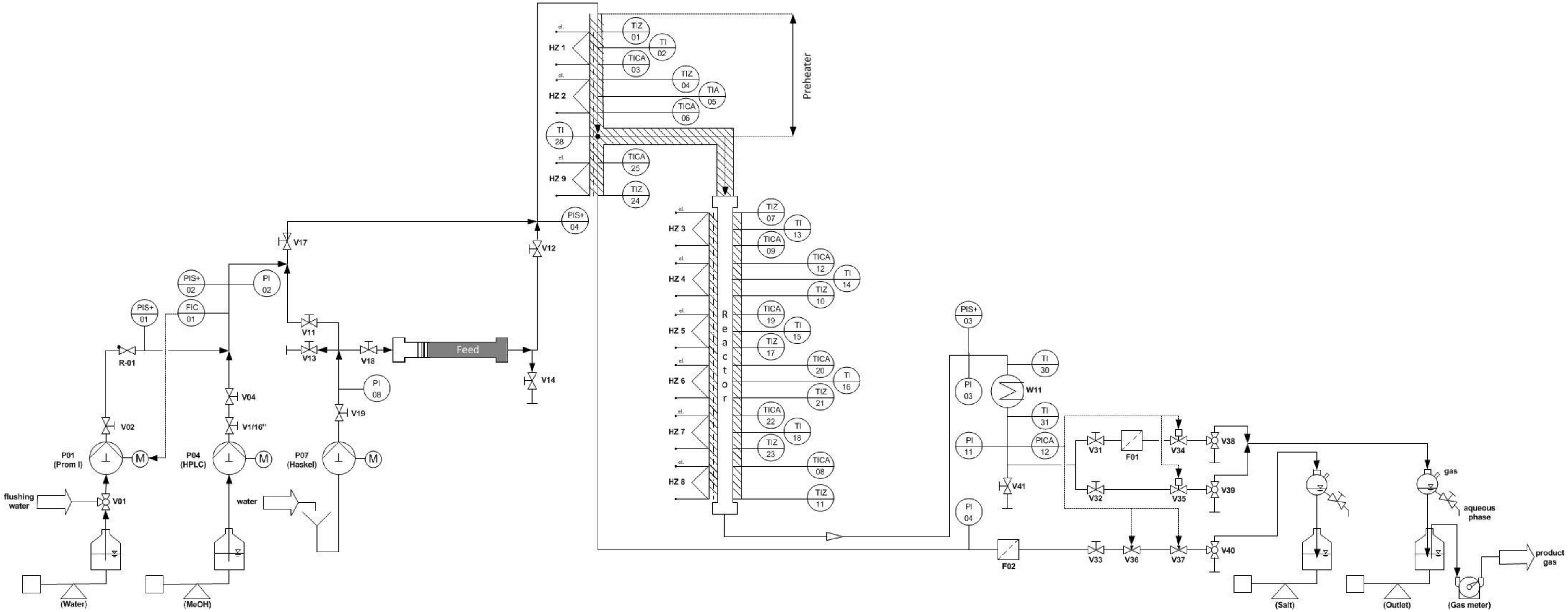


Figure 3: LENA test rig.

Figure 4 shows a simplified schematic diagram of the apparatus containing all relevant inputs and outputs. There is one input (1. Feed + H2O) and five outputs at which the gaseous, liquid and solid products of the supercritical gasification accumulate (2. Salt concentrate – 6. Filter 2 (salt concentrate)). Only at these five outputs sampling for product analysis is possible. The denotation "7. Weight Difference" in Figure 4 represents the not localizable residues in the LENA test rig after an experiment.
"6. Filter 2 (salt concentrate)" is the solid gained from the filter. "2. Salt concentrate" is the filtrated solution were soluble salts are gained.

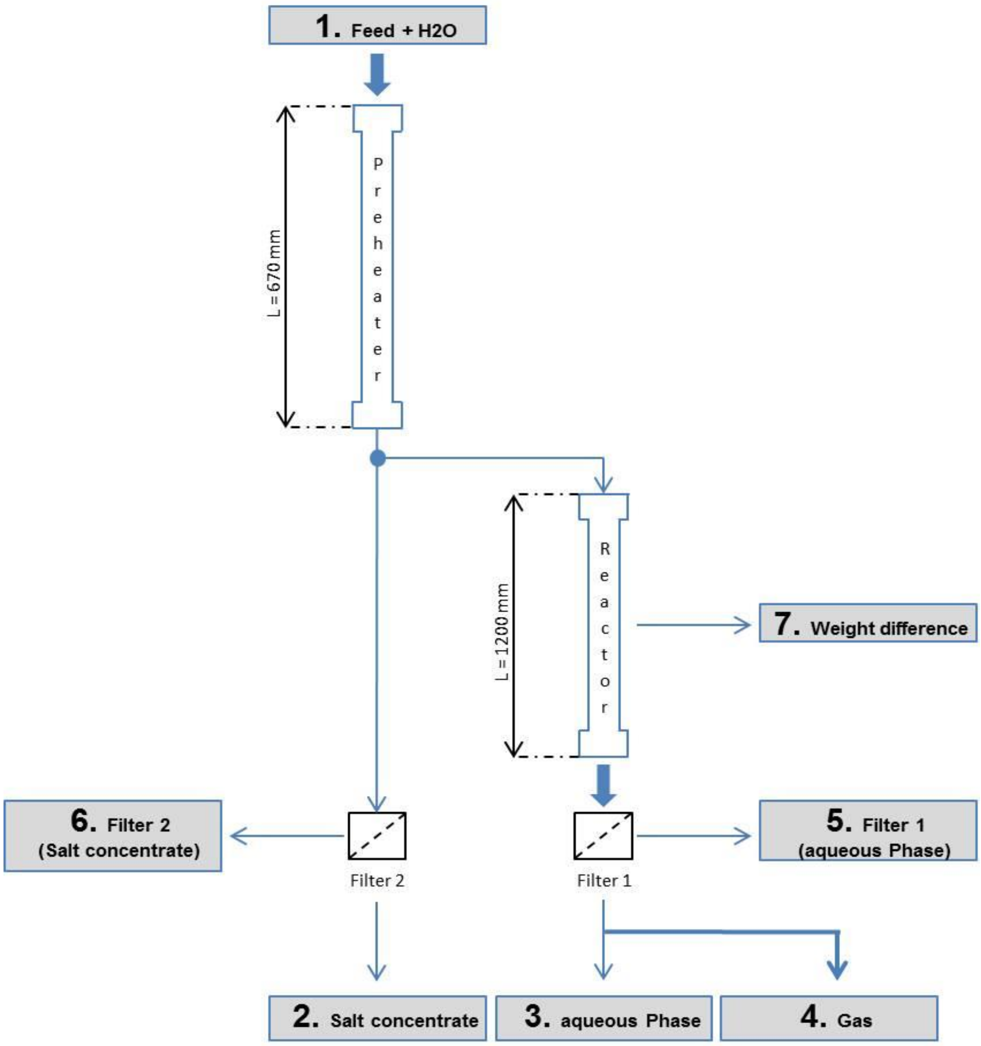


Figure 4: Schematic diagram of LENA

2.2 Pretreatment

The delivered sewage sludge samples from Lelystad and Oijen are too concentrated for hydrothermal gasification. That's why the sludge has to be diluted with a defined amount of water to receive the desired feed concentration. At first the dry matter content of the samples has to be determined. Therefore approximately 50 g sewage sludge is dried in an oven at 105 °C for 24 h. The dry matter of every charge is determined twice. Via the dry matter content the required water amount can be calculated. Moreover potassium hydrogen carbonate $KHCO_3$ is added to the feed since potassium salts catalyse the gasification reaction. To homogenize the feed a multifunctional kitchen machine is used, in which at first the required amount of dewatered sewage sludge is weighed in. Subsequent the corresponding quantity of water with diluted $KHCO_3$ is added. The whole mixture is stirred for 45 minutes and afterwards transferred into a cylindrical tank. Via the valve V14 (see Figure 3) the feed is filled into the feed tank with pressurised air.

2.3 Parameters of all experiments

All experiments were carried out at constant pressure of 280 bar because the reaction pressure until now showed only low impact on the gasification. The pilot plant is operated at 280 bar too. It is known from other feed (corn stover) that addition of K^+ enhances the gasification rate. The addition of $KHCO_3$ in experiment STOWA 1 was 5000 $\frac{\mathrm{mg\,K^+}}{\mathrm{l}}$ sludge while in all further experiments an amount of 2500 $\frac{\mathrm{mg\,K^+}}{\mathrm{l}}$ sludge was added additionally to the starting K value of the sludge. Due to different DM content the amount of K^+ per g DM is not constant, but varies with a factor of up to 2. One exception is the experiment STOWA 14 were no $KHCO_3$ was added. In Table 1 the parameters of all experiments are summarized. The selection of the experimental parameters is based on the gasification behaviour of other feeds (corn stover).

The experimental design has been fine-tuned after each experiment in order to reach stable operation and high conversion rate. Two types of sludge were investigated. The reactor temperature was varied in the range of 608 to 655 °C, the dry matter was varied between 8,59 and 17,37 wt-% and residence times between 1,7 and 6,15 min were realised. To optimize the salt separation different temperatures at the preheater from 400 to 500 °C were set.

Table 1: Experimental parameters of the lab-scale tests (constant pressure: 280 bar)

No.	Type	Dry Matter of the Feed	$T_{Preheater}$*	$T_{Reactor}$	Reactor-residence time	Feed
			(set point)	(midpoint)		
[-]	[-]	[Wt-%]	[°C]	[°C]	[min]	[g/h]
1	Lelystad	15,4	500	608	5,83	145
2		12,0	450	609	3,82	213,5
3		13,4	450	649	5,39	211,7
4		15,9	450	651	6,15	186
5		12,5	450	653	2,72	424,8
7		12,3	480	652	2,78	417,5
8		12,6	480	619	2,76	412
9		11,7	480	653	2,75	423
10	Oijen	12,7	470	649	2,76	421,1
11		12,0	460	655	1,7	701
12		12,4	450	652	5,31	215,2
13		8,59	450	653	2,92	420,8
14		12,8	460	654	2,7	430,5
15		13,4	400	653	2,66	430
16		15,0	450	653	2,75	424
17		17,4	460	648	5,43	211,2

* $T_{Preheater}$ is measured at the height of 600 mm of the Preheater from top to bottom.

In Table 2 the different charges of sewage sludge used for the lab scale-experiments are listed. Table 3 shows the duration of storage (at 3 °C), while in Table 4 the analytical results of the sludge charges are compared.

Table 2: Charges of sludge samples.

Sewage sludge	Charge - Nr.	Delivery date	DM% original	Experiments with sewage sludge charge
Type 1 LELYSTAD	1	21.02.2014	17,56	STOWA1, STOWA2
	2	21.03.2014	16,73	STOWA3, STOWA4, STOWA5
	3	29.04.2014	16,02	STOWA6, STOWA7
	4	20.05.2014	17,25	STOWA8, STOWA9
	9	16.09.1014	15,81	no experiments
Type 2 OIJEN	5	11.06.2014	24,74	STOWA10, STOWA11, STOWA12
	6	03.07.2014	27,84	STOWA13, STOWA14
	7	18.07.2014	27,03	STOWA15; STOWA16
	8	16.09.2014	31,64	STOWA17

Table 3: Storage duration of the sludge charges (The sewage sludge was stored at 3 °C and the amount that was prepared for one experiment was stored 24 h before the experiment at room temperature).

Experiments	Date of experiment	Storage time of sewage sludge	Sewage sludge	
			Charge-Nr.	Type
STOWA1	11.03.2014	18 days	1	LELYSTAD
STOWA2	19.03.2014	26 days	1	
STOWA3	26.03.2014	5 days	2	
STOWA4	03.04.2014	13 days	2	
STOWA5	10.04.2014	20 days	2	
STOWA6	05.05.2014	7 days	3	
STOWA7	16.05.2014	18 days	3	
STOWA8	22.05.2014	2 days	4	
STOWA9	05.06.2014	16 days	4	
STOWA10	13.06.2014	2 days	5	OIJEN
STOWA11	18.06.2014	7 days	5	
STOWA12	02.07.2014	22 days	5	
STOWA13	10.07.2014	7 days	6	
STOWA14	17.07.2014	14 days	6	
STOWA15	22.07.2014	4 days	7	
STOWA16	30.07.2014	12 days	7	
STOWA17	19.09.2014	3 days	8	

Table 4: Comparison of analytical results of all sewage sludge charges.

	Lelystad				Oijen			
Parameter	Charge 1 after addition of 2500 mg K^+/L	Charge 2 after addition of 2500 mg K^+/L	Charge 3 before addition of KHCO3	Charge 4 before addition of KHCO3	Charge 5 before addition of KHCO3	Charge 6 before addition of KHCO3	Charge 7 before addition of KHCO3	Charge 8 before addition of KHCO3
	[mg/g]							
TC	418	427	433	434	415	422	402	424
TIC	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TOC (C)	418	427	433	434	415	422	402	424
H	60,3	60,9	62,9	63,7	60,5	60,9	60,6	64,3
N	63,4	68,6	73	72,8	42,2	48,2	47,4	44,3
P	27,8	29,6	30,8	31,6	24	20	21,8	20,4
S	7,2	7,6	7,32	11,1	7,8	n.d.	8,7	6,68
Ca	11,7	11,1	12,7	14	22,5	18,5	16,3	13,9
K	29,6	27,8	11,8	11,4	3,6	3,2	2,96	2,82
Mg	5,89	6,4	7,02	7,2	3,1	3,1	2,86	2,61
Na	1,07	1,22	1,25	1,05	0,88	1	0,84	0,67
Si	25,7	21,8	21,6	26,3	26,5	40,6	35,3	15,79
Al	5,57	4,68	5,21	5,6	12,2	12,5	12,2	9,31
	[µg/g]							
As	8	7	7	n.d.	n.d.	n.d.	n.d.	n.d
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	138	143	153	180	5600	40	590	450
Pb	35	18	12	n.d.	110	n.d.	111	50
Zn	334	313	325	320	860	900	960	480
Cr	22	20	18	n.d.	39	150	42	20
Fe	5950	4410	3870	3800	13800	13600	20900	18500
Mo	5	6	6	n.d.	n.d.	n.d.	15	8
Ni	14	13	12	n.d.	n.d.	n.d.	24	14
Cl	584	546	670	572	1910	2020	2830	2110
Hg	0,36	0,49	0,55	0,3	0,3	n.d.	0,63	n.d.
	[J/g]							
HHV	19067	18950	18601	19161	17302	17426	16504	18105

The value 29,6 mg potassium per 1g DM of charge 1 was measured after the addition of 2500 mg K^+/L sludge, which corresponds to 20,8 mg K^+/g DM. Therefor the concentration of potassium of the original sludge must have been 8,8 mg K^+/g DM. This value is comparable to the potassium concentration of the sludge charges 3 (11,8 mg K^+/g DM) and 4 (11,4 mg K^+/g DM). In experiment STOWA 1 (charge 1 was used) 5000 mg K^+/L was added which corresponds to an addition of 31 mg K^+/g DM. The difference of the sample of charge 1 for STOWA 2 with the addition of 20,8 mg K^+/g DM compared to the sample of charge 1 for STOWA1 with the addition of 31 mg K^+/g DM is 10,2 mg K^+/1g DM. So the concentration of potassium in experiment STOWA 1 before addition of KHCO3 was 19,4 mg K^+/g DM and after the addition of $KHCO_3$ 50,4 mg K^+/g DM.

2.4 *Detailed description and results of the experiments*

The flow rate set at pump P01 or P04 is calculated to result the predetermined residence time. Thereby the following assumptions are made:

- The sewage sludge consists of dry matter and water, but for simplification it is assumed, that the density of water under operating conditions equates to the density of the sewage sludge under operating conditions.
- The overall density in the reactor is not affected by the occurring gasification reactions.
- Below 600 °C no gasification reaction takes place. The residence time was determined for a corresponding section of the reactor where the temperature was 600 °C and higher. Three temperature zones (≥600 °C, ≥630 °C and ≥660 °C) were set and for each the residence time was accurately calculated. The sum of each residence times of the temperature zones corresponds to the residence time of the reactor.

Thus the residence time of the lab-scale reactor was calculated by the following equation:

$$\tau_{reactor} = \frac{V_{reactor}}{\dot{V}_{Feed}} = \frac{V_{reactor} * \rho_{H2O}(T)}{\dot{m}_{Feed}}$$

The balance of the different elements for each experiment is evaluated on two different ways that are time dependent. One balance is formed under constant operating conditions and does not include the filters F01 and F02 (see Figure 3). Most important results of this calculation are the TOC content (TOC = total organic carbon; analysis method see Appendix) of the waste water, the gas composition and the carbon gasification yield. The second balance is a total balance that also covers the cleaning phase. Figure 5 shows the different phases of an experiment, the ethanol (EtOH)-, feed- and cleaning phase. The balance under constant operating conditions is performed at the end of the feed phase, during steady state operation (feeding rate, gas formation $\dot{V}_{Gas}$ and the gasification yield Y_{GAS} are constant).
As a preliminary test ethanol (EtOH) is hydrothermally gasified (EtOH-phase). This serves for technical survey of the LENA test rig.

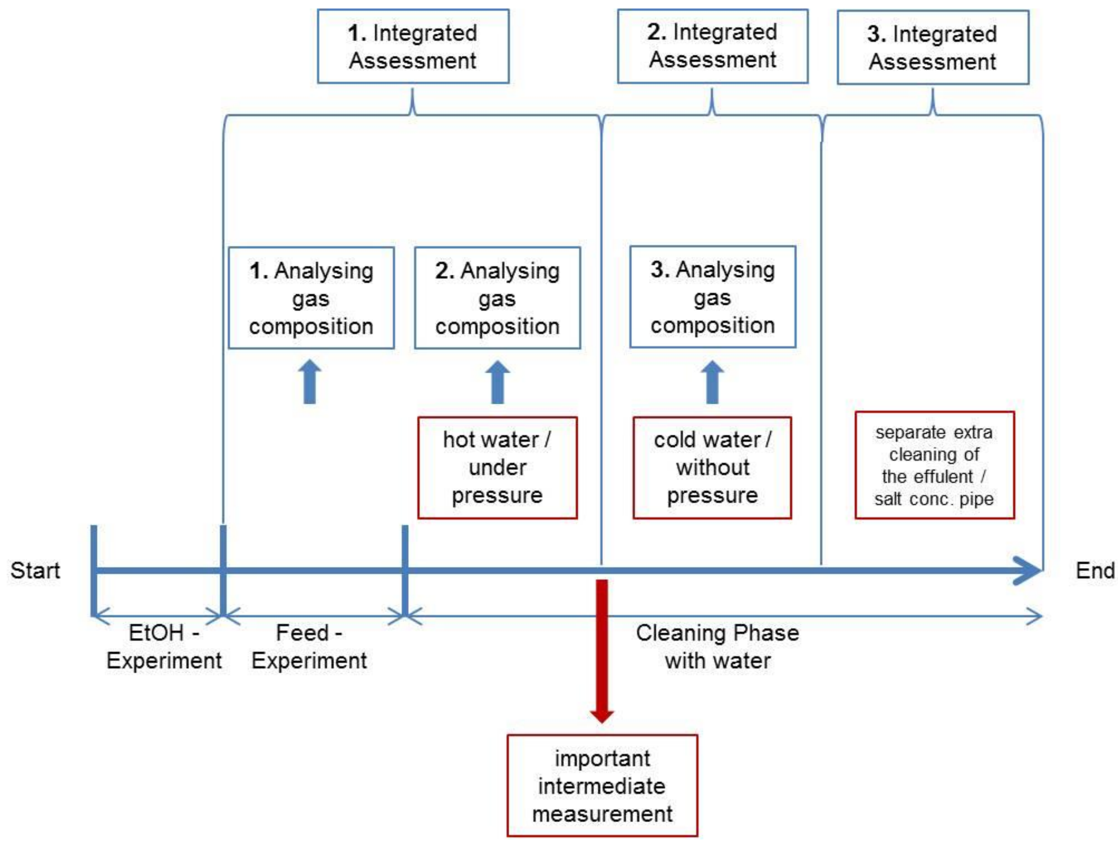


Figure 5: Balance formation during constant operating conditions and total balance.

Figure 5 not only shows the various phases of an experiment but also the structure of the calculation of the total balance. During the operation with feed the gaseous phase is analysed every 30 minutes (1. Analysing gas composition). Also the liquid phase of the effluent and of the salt concentrate is analysed. Samples of the liquid phases were usually taken every hour. Depending on the duration time of the test several samples were taken. If the experimental period allowed it at least 3 effluent samples were taken during the steady state phase of every experiment.

The cleaning phase starts with flusing the test rig with hot water under operating conditions (hot water / under pressure) while the gasification of the residual sewage sludge takes place. The formed gas is analysed (2. Analysing gas composition). Between the flushing under operating conditions and flushing with cold water (important intermediate measurement) sampling of the total effluent and the salt concentrate occured. This means that each of the two collection flasks where the total effluent and salt concentrate produced during the test are exchanged against two empty flasks for the cold cleaning phase. The total effluent and the total salt concentrate are then each sampled as one total sample. After removal of the liquid phases the flushing is carried out with cold water (cold water / without pressure). The heaters HZ1 – HZ9 are switched off and the test rig is cooled to ambient temperature. During the cleaning phase with cold water the gas phase is analysed (3. Analysing gas composition). The filter contents of F01 and F02 are analysed after the cleaning phase.

In the following the equations that were used for evaluation of the lab-scale tests are specified:

Conversion of gasification

$$Y_{Gas} = \frac{\dot{m}_{TC,Gas} * \dot{m}_{TC\ of\ dissolved\ CO2\ in\ H2O}}{\dot{m}_{TC,Feed}} * 100$$

$$\dot{m}_{TC,Gas} = \dot{m}_{C,CH4} + \dot{m}_{C,CO2} + \dot{m}_{C,CO} + \dot{m}_{C,C2H4} + \dot{m}_{C,C2H6} + \dot{m}_{C,C3H6} + \dot{m}_{C,C3H8}$$

$$\dot{m}_{TC\ of\ dissolved\ CO2\ in\ H2O} = \frac{M_C}{M_{CO_2}} \cdot (\dot{m}_{effluent} + \dot{m}_{salt\ concentrate}) \cdot S_{CO_2/H_2O}$$

$$\dot{m}_{TC,feed} = \dot{m}_{Feed-DM} * c_{TC,Feed-DM}$$

This means that carbonates, formed from CO_2 after gasification reaction, are not considered. This is important to understand the lower carbon gasification yields, but it is not correct to add CO_2 bounded as solid salts to the gasification rate.
The phase separation during the lab-scale tests takes place under low pressure. The dissolved CO_2 in water is considered in the calculation of the gasification conversion Y_{Gas}.

Y_{Gas}: Conversion of the gasification
TC: Total Carbon
$\dot{m}_{TC,Gas}$: TC-mass flow of the product gas stream
$\dot{m}_{TC\ of\ dissolved\ CO2\ in\ H2O}$: TC-mass flow of the carbon of CO2 dissolved in the effluent and in the salt concentrate
$\dot{m}_{TC,Feed}$: TC-mass flow of the feed
M_C: Molar mass of carbon
M_{CO_2}: Molar mass of CO_2
$\dot{m}_{effluent}$: mass flow of the effluent
$\dot{m}_{salt\ concentrate}$: mass flow of the salt concentrate
S_{CO_2/H_2O}: Solubility of CO_2 in H_2O
$c_{TC,Feed-DM}$: concentration of TC in the dry matter of the feed
$\dot{m}_{Feed-DM}$: dry matter mass flow of the feed

C-balance (Y_C):

$$Y_C = \frac{\dot{m}_{TC,out}}{\dot{m}_{TC,Feed}}$$

$$\dot{m}_{TC,out} = \dot{m}_{TC,effluent} + \dot{m}_{TC,salt\ concentrate} + \dot{m}_{TC,Gas}$$

$\dot{m}_{TC,out}$: TC-mass flow of all output streams
$\dot{m}_{TC,effluent}$: TC-mass flow of the effluent
$\dot{m}_{TC,salt\ concentrate}$: TC-mass flow of the salt concentrate

Cold gas efficiency (CGE):

$$CGE = \frac{TP_{total\ gas}[W]}{TP_{feed}[W]}$$

$$TP_{total\ gas} = \sum TP_i$$

$$TP_i = \dot{m}_i * HHV_i$$

$$TP_{feed} = \dot{m}_{Feed-DM} * HHV_{DM}$$

$TP_{total\ gas}$: thermal power of total gas
TP_{feed}: thermal power of the feed
TP_i: thermal power of the gas component i
$\dot{m}_i$: mass flow of the gas component i
HHV_i: higher heating value of the gas component i
HHV_{DM}: higher heating value of the dry matter of the feed

TOC-conversion:

$$TOC - conversion = 100 - \frac{\dot{m}_{TOC,liquid}}{\dot{m}_{TOC,feed}} * 100$$

$$\dot{m}_{TOC,liquid} = \dot{m}_{TOC,effluent} + \dot{m}_{TOC,salt\ concentrate}$$

$$\dot{m}_{TOC,feed} = \dot{m}_{Feed-DM} * c_{TOC,Feed-DM}$$

$\dot{m}_{TOC,out}$: TOC-mass flow of all liquid output streams
$\dot{m}_{TOC,Feed}$: TOC-mass flow of the feed
$\dot{m}_{TOC,effluent}$: TOC-mass flow of the effluent
$\dot{m}_{TOC,salt\ concentrate}$: TOC-mass flow of the liquid salt concentrate
$c_{TOC,Feed-DM}$: concentration of TOC in the dry matter of the feed

In Table 5 the analysed parameters are listed.

Table 5: Analysed parameters

Feed: dried sludge	Gas composition	Aq. effluent and salt concentrate (aq. phase)
Dry matter content	H_2	
Organic matter content	CO	
Heating value	CO_2	
CHN(O) analysis	CH_4	
TC	C_2H_4	TC
TIC	C_2H_6	TIC
TOC	C_3H_6	TOC
H	C_3H_8	P
P		S
S		PO4
N		SO4
Ca		TNb
K		NO_3 and NO_2, NH_4^+
Mg		Ca
Na		K
Si		Mg
Al		Na
As		Si
Cd		Al
Cu		As
Pb		Cd
Zn		Cu
Cr		Pb
Fe		Zn
Mo		Cr
Ni		Fe
Cl		Mo
Hg		Ni
		Cl
		Hg
		Phenol Index

The composition of the solids is determined by microwave digestion and subsequent ICP-OES Analysis. The limit of quantification of solids (see Appendix) is dependent from the weight of sample taken and from the digestion volume. Also energy dispersive X-ray spectroscopy coupled with a scanning electron microscope of the filter and other solid residues is performed.
The liquid samples are analysed by high-performance liquid chromatography (HPLC). The concentration of total carbon TC and total inorganic carbon TIC is determined by combustion of the liquid samples and measurement of carbon dioxide. Via the difference of TC to TIC the concentration of total organic carbon TOC is calculated. The concentration of total nitrogen bonded TN_b is determined by combustion of the liquid samples in a nitrogen atmosphere (analysis method see appendix). The determination of the elements $P, S, Al, Pb, Cd, Ca, Cr, Fe, K, Cu, Ca, Mg, Mo, Na, Ni, Si, Zn$ and Hg is carried out by Optical Emission Spectroscopy with inductively coupled plasma (ICP-OES). With the liquid phase no digestion took place before ICP. The liquid phase was only filtrated and the aqueous phase was analysed. As (Arsen) and partially some other elements are measured by atomic absorption spectroscopy (AAS) with a detection limit being smaller by a factor of 1000 compared to ICP-OES. The limit of quantification of liquids (see Appendix) is dependent from the dilution factor.
The gaseous samples are analysed by gas chromatography (GC) with a thermal conductivity detector (TCD) and a flame ionization detector (FID). Additionally GC-MS is performed for few samples. Calibration of the GC is done with a gas which consists of H_2, CH_4, C_2H_6, CO, CO_2, C_2H_4, C_3H_8, C_3H_6.

Table 6 shows the carbon balance for all experiments under operating conditions. The balance period is selected according to constant gas production $\dot{V}_{Gas}$ and gasification yield Y_{Gas}. $Feed_{total}$ is the total pumped amount of sewage sludge within the duration of the balance period. C_{total} is the total amount of carbon in $Feed_{total}$. Thus the TOC and TIC amount of the feed as well as the carbon input via $KHCO_3$ is taken into account. The TOC-, NH_4^+-, TN_b-values of the effluent water are averaged values from a few individual measurements. After the hydrothermal gasification the TIC yield increases in all experiments. The carbon yield Y_C specifies the outflow of carbon via the gaseous and liquid phase related to the inflow of carbon during constant operating conditions (residues in the filters F01 and F02 and further accumulation areas within the apparatus are not taken into account).

Table 6: Results of the steady-state phase

No. [-]	Phase time[1] [h]	$Feed_{total}$ [g]	C_{total} [g]	Plugging [after h]	Y_{Gas} [%]	Y_C [%]	TOC-destruction [%]	Y_{TIC} [%]	Cold gas efficiency η [-]	TOC- waste water [mg/l]	NH_4^+- waste water [mg/l]	TNb-waste water [mg/l]
1	2,75	398	26,4	2,75	27,9	29,5	99,2	-93	0,38	-	-	-
2	5,50	1174	59,9	No	37,9	51,2	92,8	308	0,43	2565	9208	6724
3	3,00	635	36,9	No	52,9	66,1	96,8	655	0,62	1870	12674	7715
4	0,50	93	6,4	3,25	33,0	46,5	90,8	229	0,38	-	-	-
5	2,52	1069	58,1	4,85	63,8	80,9	94,9	750	0,71	3415	13113	7038
7	4,00	1670	90,4	No	64,0	77,9	97,3	688	0,72	1725	13012	7243
8	3,00	1236	68,8	No	49,9	65,8	93,4	570	0,50	3072	11800	6580
9	5,00	2115	109,2	No	68,0	80,7	97,8	616	0,77	1953	11724	9300
10	3,93	1657	88,9	6,50	71,2	81,8	96,7	342	0,87	2141	8821	5580
11	2,78	1402	71,1	No	71,3	84,2	94,9	348	0,91	2363	8456	5591
12	2,50	538	28,2	No	75,7	86,0	97,9	461	0,93	1154	9796	6845
13	4,00	1683	62,5	No	77,3	87,1	97,9	270	0,95	841	6645	3805
14	2,00	861	47,3	2,3 / No[2]	63,4	75,1	94,7	288	0,65	2817	6196	5820
15	3,00	1290	71,5	6,25	65,3	78,7	95,6	544	0,75	2534	6548	7771
16	2,00	846	52,5	No	75,9	81,6	96,5	196	0,83	2392	12413	7364
17	2,00	421	31,3	No	48,6	58,3	97,2	564	0,60	800	10380	6379

[1]duration of the steady state phase

[2]The experiment was interrupted as a plugging occurred and was then restarted.

2.4.1 Experiment STOWA 1

The selection of the experimental parameters for the first experiment was based on the gasification behaviour of other feeds (corn stover). Due to a blockage of the flow during the feed phase the experiment STOWA 1 proceeded irregular and finally had to be stopped after 2,75 h. The plugging was localized in the brine separation system, 84 cm distant from the preheater. Figure 6 shows the black and grey porous particles of different size (max. diameter 12 cm) which plugged the pipe. The composition of the particles was determined by EDX (energy dispersive X-ray spectroscopy) (see Table 7 and Table 8). The analysis show that the particles mainly consist of carbon and oxygen. This indicates the formation of char.

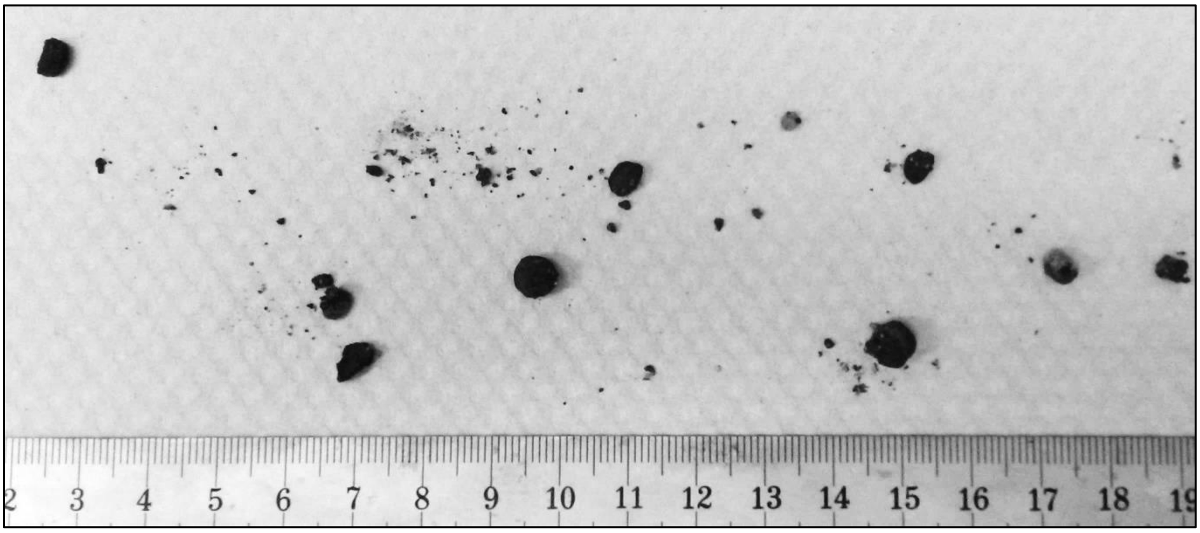


Figure 6: Residues in the brine separation system of experiment STOWA 1.

Table 7: EDX results of the black particles responsible for plugging during STOWA 1.

Element	Massen%	Atom%
C K	32.79	44.47
N K	0.00	0.00
O K	41.71	42.47
Na K	0.26	0.18
Mg K	2.57	1.72
Al K	2.46	1.49
Si K	5.08	2.95
P K	6.69	3.52
S K	0.27	0.14
K K	1.63	0.68
Ca K	4.12	1.68
Ti K	0.32	0.11
Fe K	1.81	0.53
Cu L	0.28	0.07
Insgesamt	100.00	

The letters K and L behind the elements are characteristic X-ray lines for the evaluation of the EDX analysis.

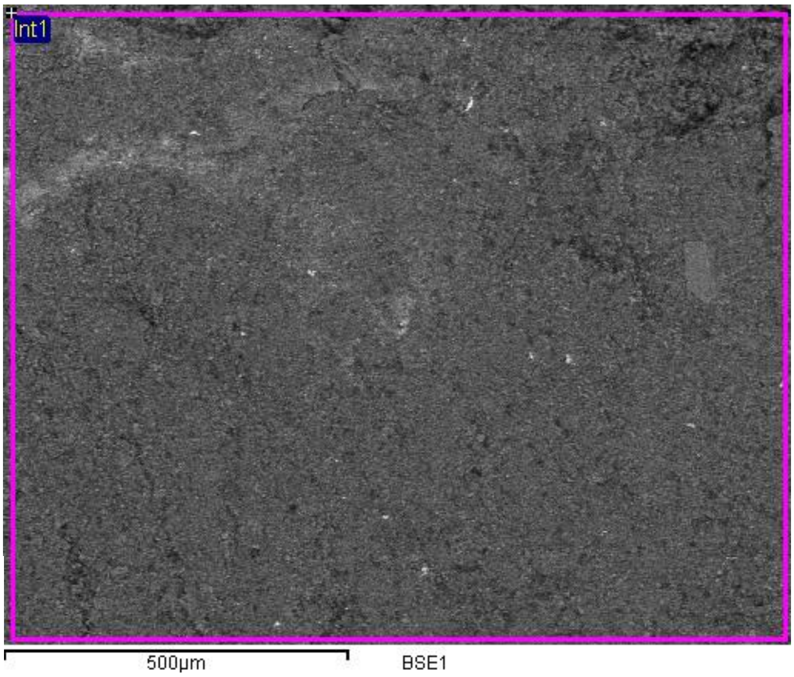
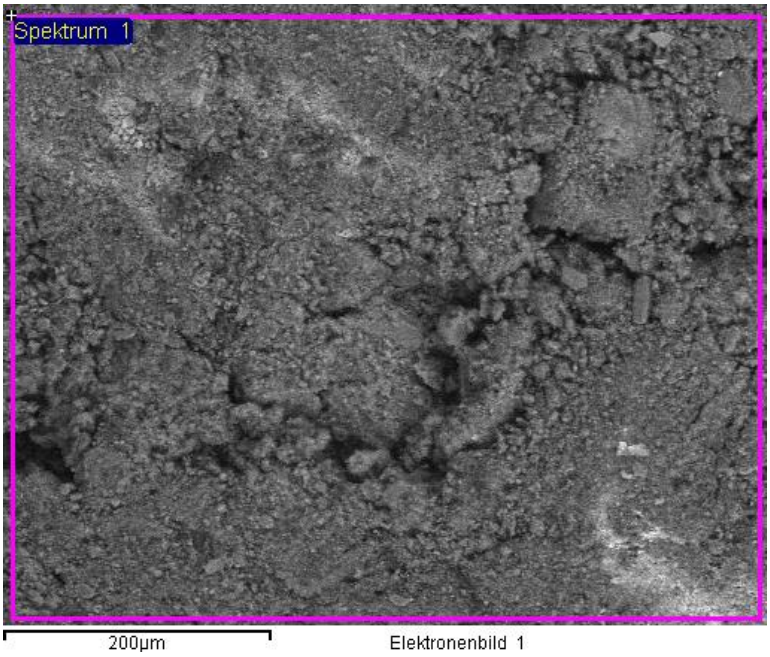


Table 8: EDX results of the greyish black particles responsible for plugging during STOWA 1.

Element	Massen%	Atom%
C K	24.84	35.98
N K	0.00	0.00
O K	43.18	46.94
Na K	0.25	0.19
Mg K	2.75	1.97
Al K	2.53	1.63
Si K	5.34	3.31
P K	9.07	5.09
S K	0.30	0.16
Cl K	0.04	0.02
K K	2.34	1.04
Ca K	5.99	2.60
Ti K	0.46	0.17
Fe K	2.92	0.91
Insgesamt	100.00	



The letter K behind the elements is a characteristic X-ray line for the evaluation of the EDX analysis.

Experiment conditions		
Dry matter content	15,44	%
K^+-conc. ($KHCO_3$)	5000	mg/l
real Temperature	608	°C
pressure	280	bar
real residence time	5,83	min
real Feed mass flow	145,0	g/h

Sewage sludge		
time	2,75	h
Total sludge fed	398,8	g
C-mass in the total feed	25,73	g
TOC-mass in the total feed*	25,73	g
Total mass effluent	319,0	g
Total mass salt brine	61,7	g
TOC-Conc.		
Effluent sample	0,64	mg/g
Salt conc. sample (Position 2)	-	mg/g
Gas production	7,17	Nl/h
Conversion of gasification (TC)	27,9	%
TOC-conversion**	99,2	%
Mass-balance	105,3	%
C-balance (TC)	29,5	%
C-deficit (TC)	-18,8	g
Gas composition		
H_2	41,06	Vol %
CO	1,38	Vol %
CO_2	21,03	Vol %
CH_4	27,36	Vol %
C_2H_4	0,08	Vol %
C_2H_6	8,90	Vol %
C_3H_6	0,03	Vol %
C_3H_8	0,16	Vol %
*TIC in the feed not detectable		
** without the C from $KHCO_3$		

The masses of filter 1 and filter 2 were determined from experiment 5 to experiment 17.

STOWA 1						
	Feed analysis		aq. Effluent analysis during experiment (Position 3)			salt concentrate analysis (Position 2)
Parameter	Concentration in dry matter	Concentration diluted sludge*	after 1,25 hours	after 1,75 hours	after experiment	after experiment
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]
TC	418	64539	162	1308		
TIC	n.a.	n.a.	100	164		
TOC (C)	418	64539	133	1143		
H	60,3	9310	-	-		
N	63,4	9789	-	-		
O	-	-	-	-		
NO3	-	-	-	-		
NO2	-	-	-	-		
NH4	-	-	-	-		
TNb	-	-	-	-		
P	27,8	4292	6,30	227		
PO4	-	-	-	-		
S	7,2	1112	-	-		
SO4	-	-	-	-		
Ca	11,7	1806	1,34	5,69		
K	39,2**	4570	25,90	1475		
Mg	5,89	909,4	-	-		
Na	1,07	165,2	-	-		
Si	25,7	3968	-	-		
Al	5,57	860,0	n.d.	n.d.		
As	0,008	1,24	-	-		
Cd	n.a.	n.a.	-	-		
Cu	0,138	21,3	-	-		
Pb	0,035	5,40	-	-		
Zn	0,334	51,6	-	-		
Cr	0,022	3,40	n.d.	n.d.		
Fe	5,95	918,7	0,19	0,02		
Mo	0,005	0,772	n.d.	n.d.		
Ni	0,014	2,16	n.d.	n.d.		
Cl	0,584	90,17	-	-		
Hg	0,00036	0,056	-	-		
Phenolindex	-		-	-	317 1)	291 1)

* for simplification it is assumed that the density of water equates to the density of sewage sludge
** detected after addition of $KHCO_3$

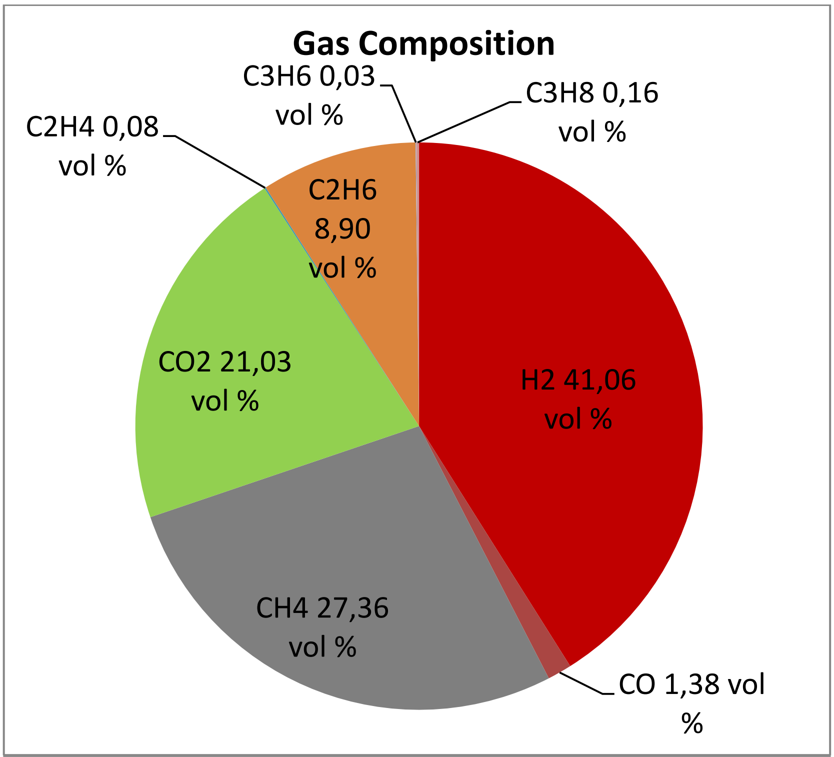
Legend:

1)	Values of the filter water
n.d.	not detectable or signal interference

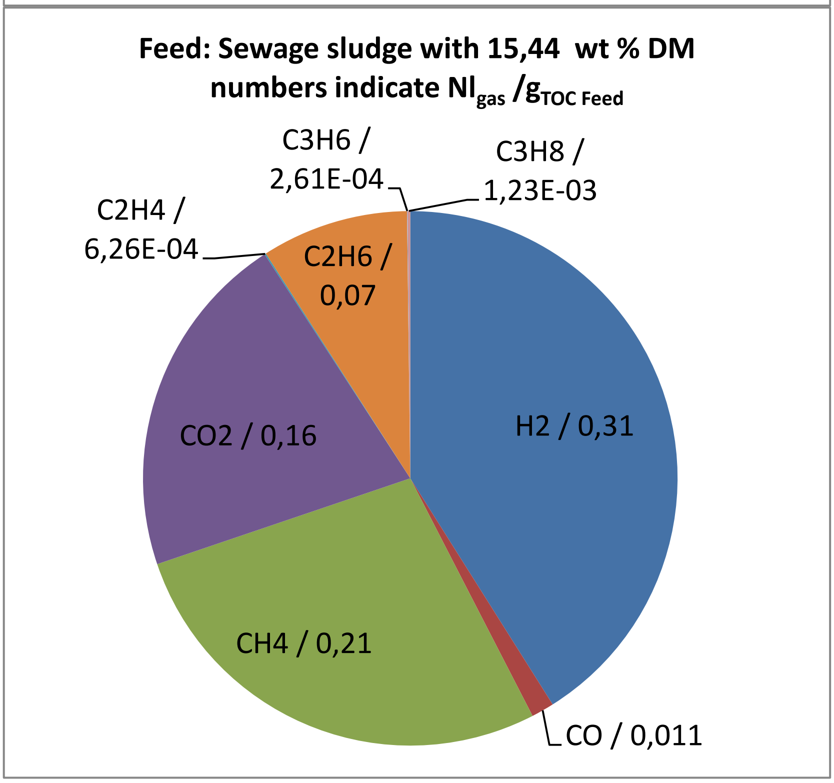
The values of the column "after 1,75 hours" are mostly higher than the values of the column "after 1,25 hours" because the effluent after 1,25 hours still arises from the feeding of the ethanol water mixture. After 1,75 hours the effluent of the gasification of sewage sludge exits the plant.

Analysis sewage sludge from Lelystad sample 21.2.14

Feed			
HHV:		19067	J/g
Ash content	by 550°C:	19,94	%
	by 815°C:	18,65	%
	by 1000°C:	18,52	%



Gas Composition
C3H6 0,03 vol %
C3H8 0,16 vol %
C2H4 0,08 vol %
C2H6 8,90 vol %
CO2 21,03 vol %
H2 41,06 vol %
CH4 27,36 vol %
CO 1,38 vol %



Feed: Sewage sludge with 15,44 wt % DM
numbers indicate Nl$_{gas}$ /g$_{TOC\ Feed}$
C3H6 / 2,61E-04
C3H8 / 1,23E-03
C2H4 / 6,26E-04
C2H6 / 0,07
CO2 / 0,16
H2 / 0,31
CH4 / 0,21
CO / 0,011

2.4.2 Experiment STOWA 2

In comparison to the first experiment the preheater temperature and the feed concentration was lowered to avoid plug in the preheater. Experiment STOWA 2 ran stable. After the experiment black and greyish black particles were found in the preheater and were analysed by EDX. The compositions of the particles are listed in Table 9 and Table 10. The analysis show that the particles mainly consist of carbon and oxygen.

Table 9: EDX results of the black particles found in the preheater after experiment STOWA 2.

Element	Massen%	Atom%
C	42.45	56.05
O	32.91	32.62
Na	0.09	0.06
Mg	0.40	0.26
Al	0.41	0.24
Si	10.29	5.81
P	2.11	1.08
S	1.41	0.70
K	1.58	0.64
Ca	1.70	0.67
Ti	0.09	0.03
Cr	1.45	0.44
Fe	0.59	0.17
Ni	4.53	1.22
Insgesamt	100.00	

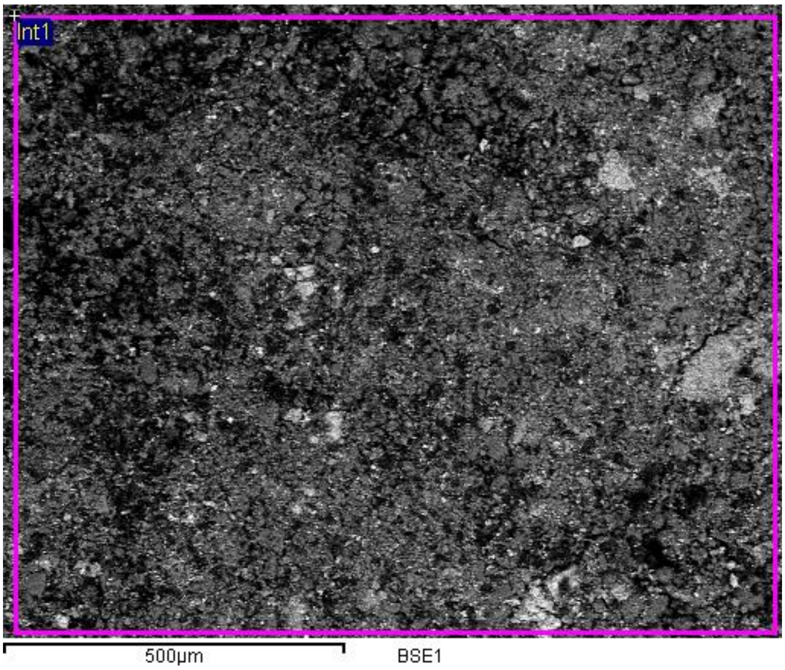
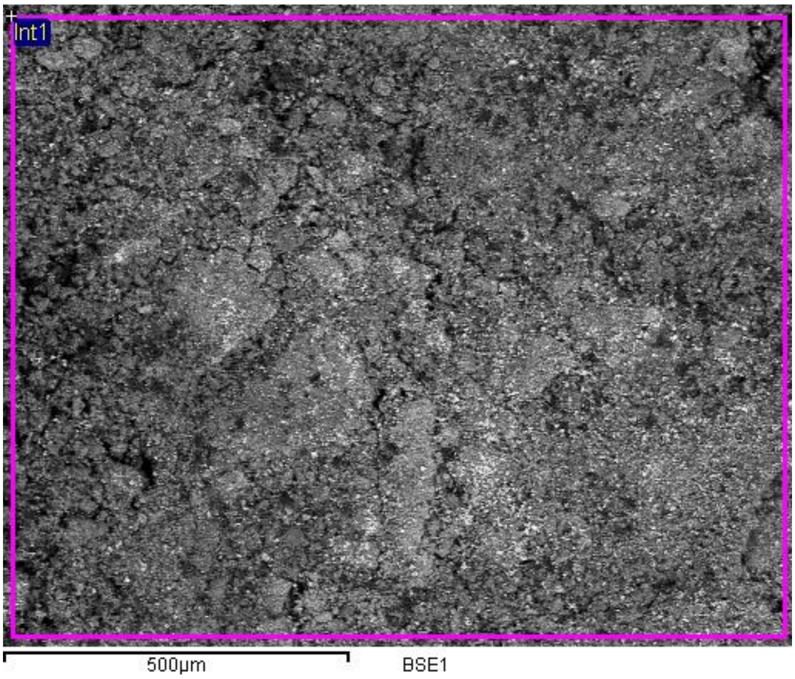


Table 10: EDX results of the greyish black particles found in the preheater after experiment STOWA 2.

Element	Massen%	Atom%
C	35.67	51.78
O	29.72	32.39
Na	0.16	0.12
Mg	0.62	0.44
Al	0.81	0.52
Si	7.21	4.48
P	3.70	2.08
S	2.67	1.45
K	3.46	1.54
Ca	2.10	0.91
Ti	0.10	0.04
Cr	3.39	1.14
Fe	1.12	0.35
Ni	9.28	2.76
Insgesamt	100.00	



The filter residues of F01 and F02 were not analysed. Only a tarry sample (5,6 g) of filter F01 was examined by GC-MS-analysis (only qualitative) . The composition was determined by comparison of the results with the database NIST 05 mass spectral library. The main components are indoles, naphthalenes, anthracenes, pyrenes and fluorenes. This is an indication for tar formation.

Experiment conditions		
Dry matter content	12,00	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	609	°C
pressure	280	bar
real residence time	3,82	min
real Feed mass flow	213,5	g/h

Sewage sludge		
time	5,5	h
Total sludge fed	1174	g
C- mass in the total feed*	58,9	g
Total mass effluent	566,0	g
Total mass salt brine	404,2	g
TOC-Conc.		
Effluent sample	2,94	mg/g
Salt conc. Sample (Position 2)	5,33	mg/g
Gas production	8,22	Nl/h
Conversion of gasification (TC)	37,88	%
TOC-conversion**	92,78	%
Mass-balance	98,14	%
C-balance (TC)	51,15	%
C-deficit (TC)	-29,21	g
Gas composition		
H_2	22,43	Vol %
CO	0,18	Vol %
CO_2	32,28	Vol %
CH_4	31,97	Vol %
C_2H_4	0,51	Vol %
C_2H_6	10,94	Vol %
C_3H_6	0,31	Vol %
C_3H_8	1,38	Vol %
*TIC in the feed not detectable ** without the C from $KHCO_3$		

STOWA 2									
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**residue of preheater**	
Parameter	Concentration in dry matter	Concentration diluted sludge*	after 5,83 hours	after 6,83 hours	after 7,83 hours	after experiment	after experiment	grey, dark pieces	dark pieces
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[weight %]	[weight %]
TC	418	50160	7784	7735	8012	5504	12709	35, 7	42, 5
TIC	n.d.	n.d.	4873	5337	5626	4414	1058	-	-
TOC (C)	418	50160	2911	2398	2387	1090	11650	-	-
H	60,3	7236	-	-	-	-	-	-	-
N	63,4	7608	-	-	-	-	-	n.d.	n.d.
O	-	-	-	-	-	-	-	29,7	32,9
NO3	-	-	n.d.	n.d.	n.d.	20,31	n.d.	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NH4	-	-	7205	9535	10883	6787	2476	-	-
TNb	-	-	5763	7114	7295	5437	2862	-	-
P	27,8	3336	28,0	45,3	54,9	59,7	937	3,7	2,11
PO4	-	-	1)	1)	1)	43,85	3152	-	-
S	7,2	864	20,7	24,8	24,5	24,7	287	2,67	1,41
SO4	-		19,9	25,4	28,5	28,4	591	-	-
Ca	11,7	1404	2,43	1,82	2,06	3,35	5,43	2,1	1,7
K	29,6**	3552	199	275	319	308	3010	3,46	1,58
Mg	5,89	707	1,17	0,97	1,14	2,48	1,56	0,62	0,4
Na	1,07	128	5,10	6,75	7,97	8,15	59,7	0,16	0,09
Si	25,7	3084	12,1	17,8	23,4	14,1	85,7	7,21	10,3
Al	5,57	668	0,24	0,19	0,30	0,16	n.d.	0,81	0,41
As	0,008	0,96	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,138	16,6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Pb	0,035	4,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,334	40,1	0,0304	0,0148	0,0482	0,0263	0,0644	n.d.	n.d.
Cr	0,022	2,64	n.d.	n.d.	n.d.	n.d.	n.d.	3,39	1,45
Fe	5,95	714	0,42	0,24	0,10	0,74	1,21	1,12	0,59
Mo	0,005	0,6	0,14	0,11	0,10	0,20	3,32	n.d.	n.d.
Ti	n.d.	n.d.	-	-	-	-	-	0,1	0,09
Ni	0,014	1,68	n.d.	n.d.	n.d.	n.d.	n.d.	9,28	4,53
Cl	0,584	70,1	22,1	21,31	21,7	22,7	41,7	n.d.	n.d.
Hg	0,00036	0,0432	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	297	353	369	264	92	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge
** detected after addition of $KHCO_3$

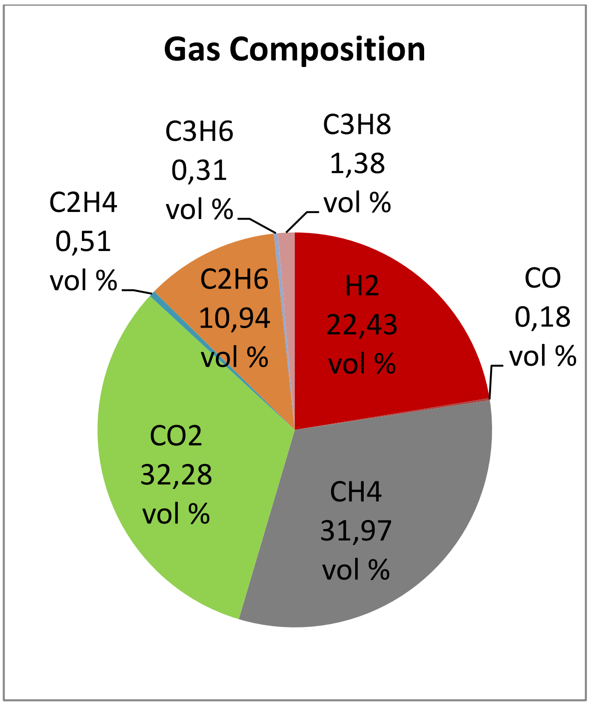
Legend	
1)	not evuable
n.d.	not detectable or signal interference

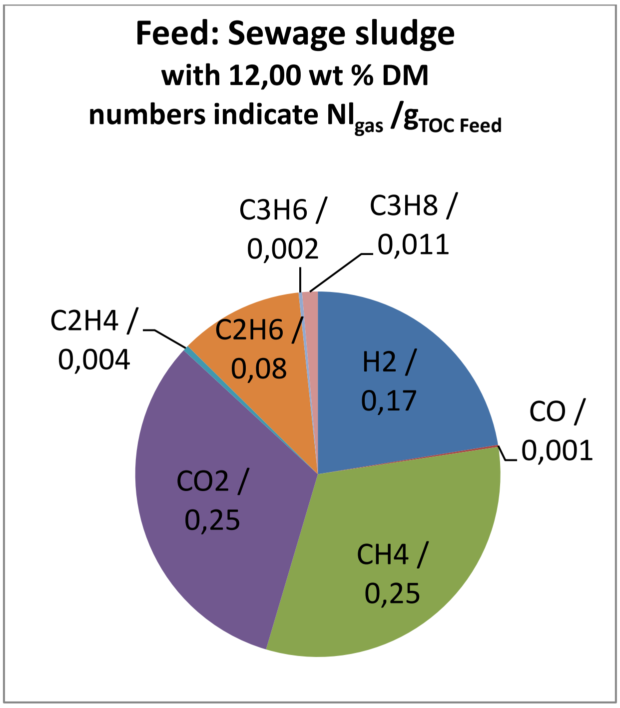
"After experiment" means that point of time after the operation with sewage sludge and the phase of flushing with water at high temperature and pressure where each one total sample of the salt concentrate and one total sample of the effluent are taken from the corresponding collection flasks. This counts analogously for all other results of the lab-scale experiments.

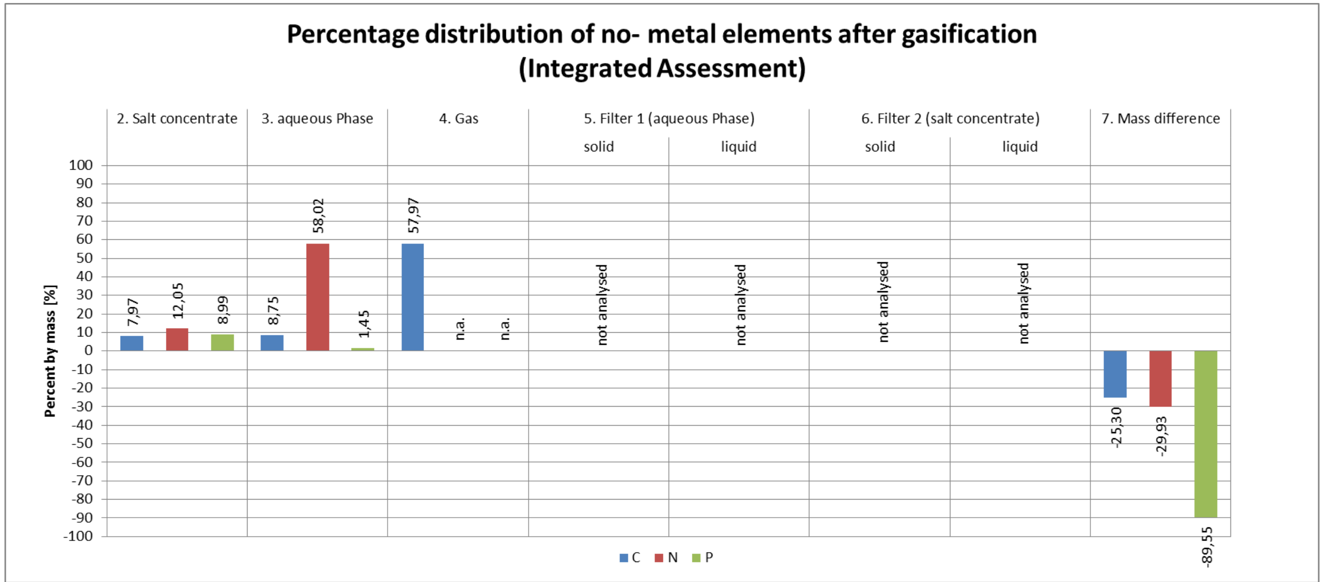
The TC and $NH4^+$ values of the effluent "after experiment" are lower than measured during the experiment, because the total effluent sample was more diluted caused by the flushing with water after feeding of sewage sludge. Otherwise for example the concentrations of P, Na and S strongly increase because these elements accumulate during the operation with sewage sludge in the apparatus and exit the plant with delay during the flushing phase with water.

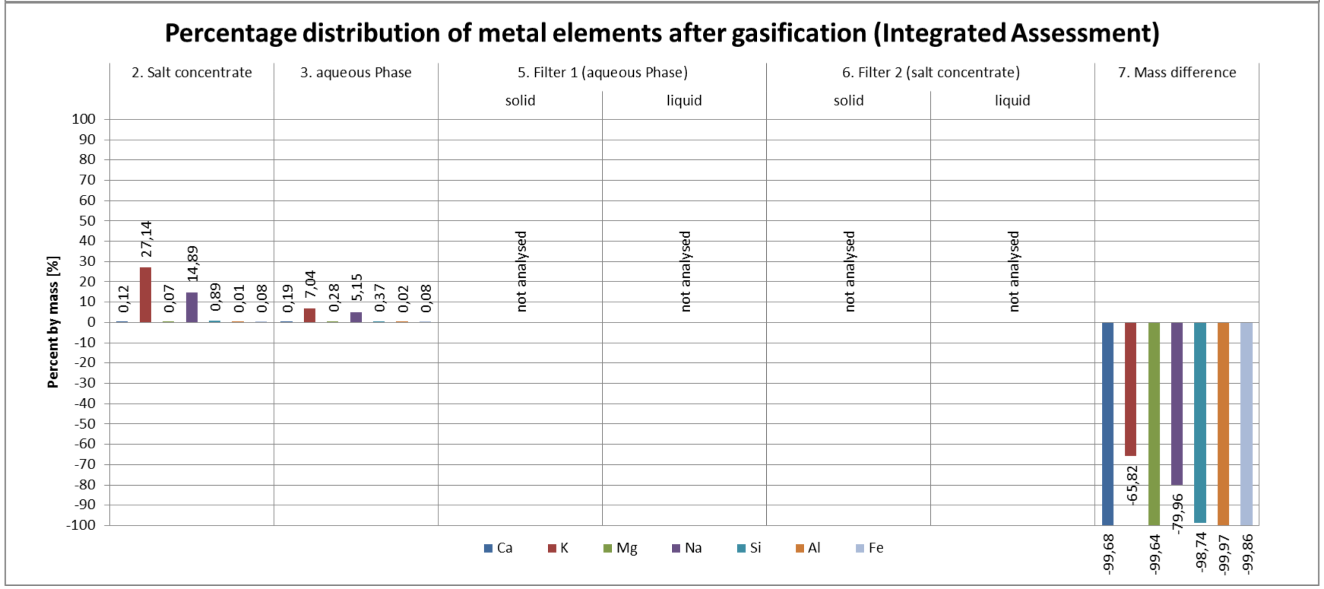
Additional analytical data: Sewage sludge from Lelystad sample 21.2.14

Feed			
HHV:		19067	J/g
Ash content	by 550°C:	19,94	%
	by 815°C:	18,65	%
	by 1000°C:	18,52	%









The cleaning phase with cold water is not incorporated in the total balance of experiment STOWA 2.

2.4.3 Experiment STOWA 3

To increase the gasification yield, the reaction temperature was increased to 649 °C. The experiment STOWA 3 ran stable.

Experiment conditions		
Dry matter content	13,42	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	649	°C
pressure	280	bar
real residence time	5,39	min
real Feed mass flow	211,7	g/h

Sewage sludge		
time	3	h
Total sludge fed	635,0	g
C- mass in the total feed*	36,4	g
Total mass effluent	417	g
Total mass salt birne	95,6	g
TOC-Conc.		
Effluent sample	1,73	mg/g
Salt conc. sample (Position 2)	2,83	mg/g
Gas production	12,8	Nl/h
Conversion of gasification (TC)	52,9	%
TOC-conversion**	96,8	%
Mass-balance	101,1	%
C-balance (TC)	66,1	%
C-deficit (TC)	12,5	g
Gas composition		
H_2	18,56	Vol %
CO	0,26	Vol %
CO_2	31,33	Vol %
CH_4	37,68	Vol %
C_2H_4	0,29	Vol %
C_2H_6	11,78	Vol %
C_3H_6	0,03	Vol %
C_3H_8	0,08	Vol %

*TIC in the feed not detectable
** without the C from $KHCO_3$

STOWA 3								Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**Filter1 (Position 5)**	**Filter2 (Position 6)**
Parameter	Concentration dry matter	Concentration diluted sludge*	after 7,00 hours	after 8,00 hours	after 9,00 hours	after experim ent	after experiment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	
TC	427	57303	7996	7924	7923	3915	8421	517	132
TIC	n.d.	n.d.	7996	7487	7923	2940	581	n.d.	n.d.
TOC (C)	427	57303	1820	1920	1870	975	7840	517	132
H	60,9	8173	-	-	-	-	-	n.d.	n.d.
N	68,6	9206	-	-	-	-	-	30,6	19,2
O	-	-	-	-	-	-	-	-	-
NO3	-	-	69	68	70	n.d.	n.d.	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NH4	-	-	12120	13377	12526	6745	1901	-	-
TNb	-	-	7661	7692	7793	3849	7150	-	-
P	29,6	3972	86,9	115	106	65	1650	44	109
PO4	-	-	313	411	401	186	5169	-	-
S	7,6	1020	63,0	164	104	55	309	14,1	3,7
SO4	-	-	54,0	62,1	68,8	60	676,5	-	-
Ca	11,1	1490	0,96	0,85	0,78	1,98	3,55	32,1	63,9
K	27,8**	3731	207	274	270	201	4275	18,8	45,2
Mg	6,4	858,9	3,92	2,88	2,38	3,73	14,8	15,3	36,3
Na	1,22	163,7	6,55	7,48	8,35	6,27	130	1,27	2,84
Si	21,8	2926	36,0	41,6	42,1	32,7	95,5	43,6	69,8
Al	4,68	628,1	n.d.	n.d.	n.d.	0,11	0,2	9,83	23,1
As	0,007	0,939	n.d.	n.d.	n.d.	n.d.	n.d.	0,015	0,007
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,143	19,2	n.d.	n.d.	n.d.	n.d.	n.d.	0,683	0,55
Pb	0,018	2,42	n.d.	n.d.	n.d.	n.d.	n.d.	0,048	0,085
Zn	0,313	42,0	n.d.	n.d.	n.d.	n.d.	n.d.	0,812	1,67
Cr	0,02	2,68	n.d.	n.d.	n.d.	n.d.	n.d.	1,27	0,422
Fe	4,41	591,8	0,21	0,05	0,05	0,10	0,60	12,3	23,5
Mo	0,006	0,805	0,04	0,02	0,03	0,00	0,23	2,39	0,2
Ni	0,013	1,745	n.d.	n.d.	n.d.	n.d.	n.d.	15,3	0,918
Cl	0,546	73,3	226	223	226	28,4	63,2	0,321	0,207
Hg	0,00049	0,066	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	262	268	264	109	60	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

** detected after addition of $KHCO_3$

Legend	
n.d.	not detectable or signal interference

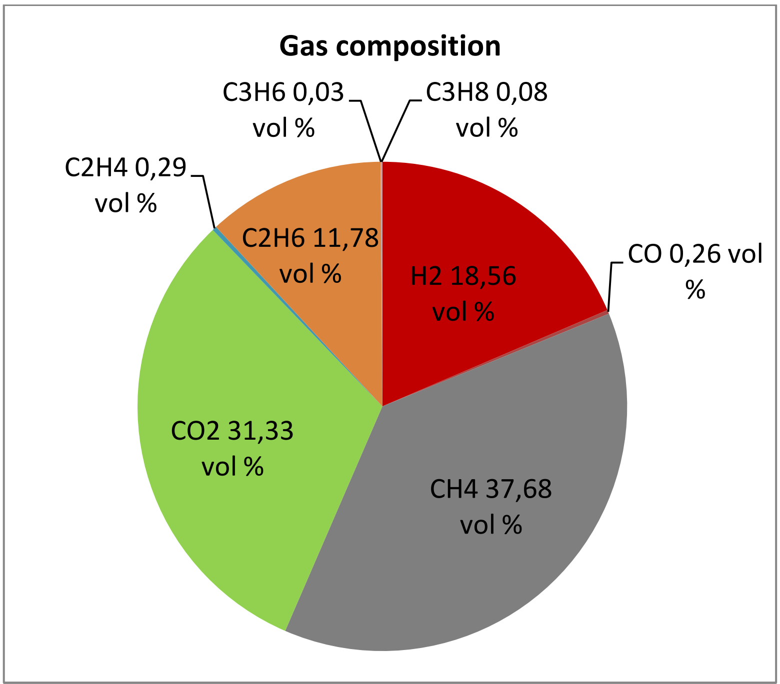
Additional analytical data: Sewage sludge from Lelystad sample 21.3.14

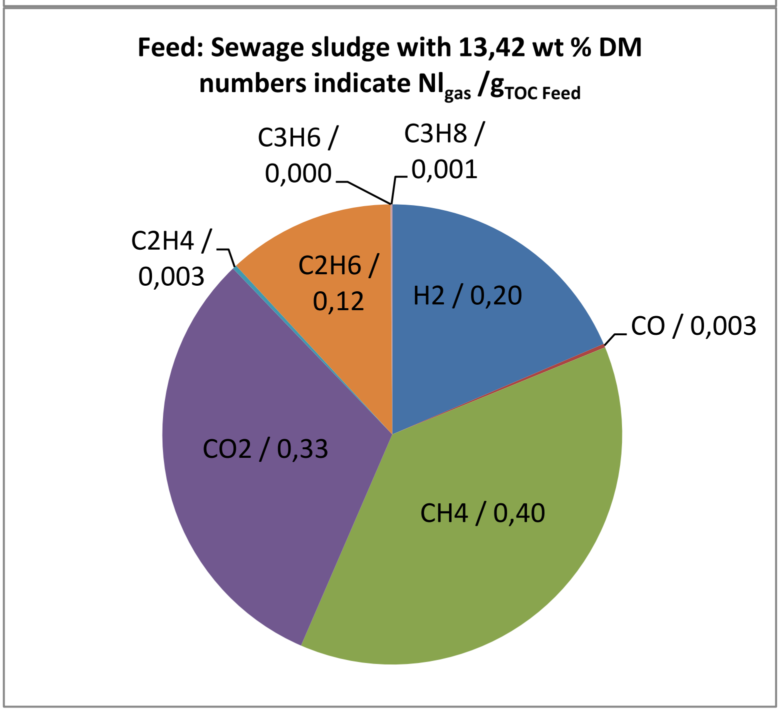
Feed			
HHV:		18950	J/g
Ash content	by 550°C:	19,4	%
	by 815°C:	18,9	%
	by 1000°C:	18,6	%

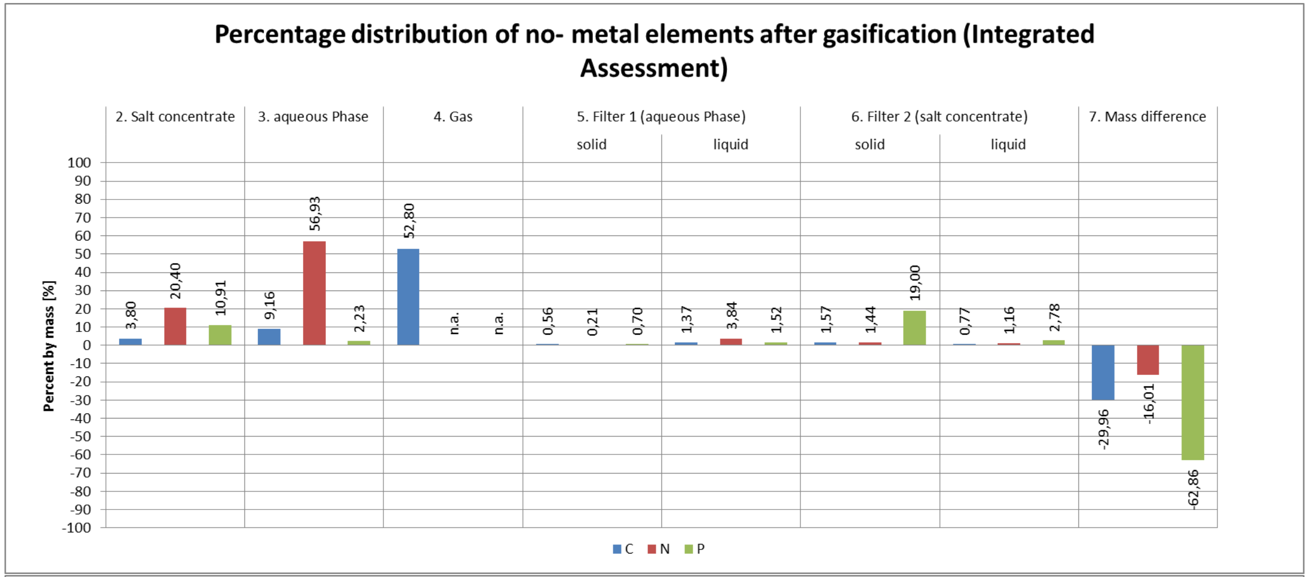
Mass of solids:

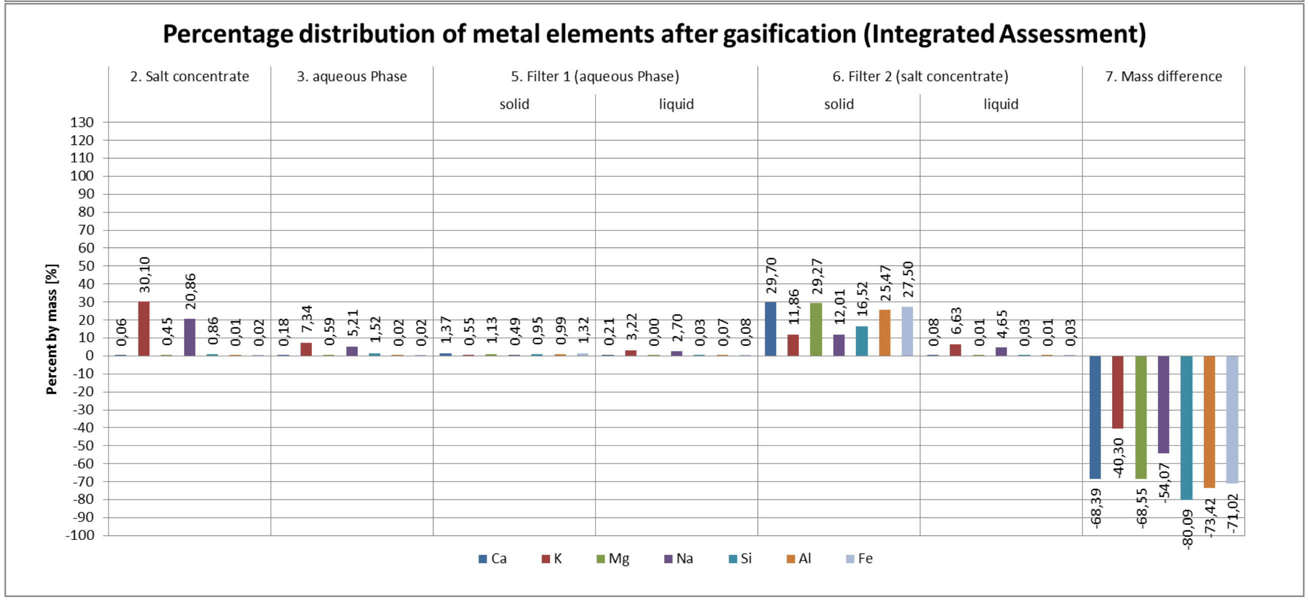
filter1 - aqueous Phase	m=	1,416	g
filter2 - salt concentrate	m=	15,45	g

Method: ICP. Determination of O only performed ba EDX









The cleaning phase with cold water is not incorporated in the total balance of experiment STOWA 3.

2.4.4 Experiment STOWA 4

In experiment STOWA 4 the longest residence time (6,15 s) of all tests was realized and a feed with a higher dry matter content (15,88 wt.-%) than in the experiments before was used. Because of a blockage of the flow rate during the feed phase experiment STOWA 4 proceeded irregular and finally had to be stopped after 3,25 h. In the preheater and the tee, for salt separation, particles were found. The compositions of these particles which caused the blockage were determined by EDX analysis (see Table 11 and Table 12). The analysis shows that the particles mainly consist of carbon and oxygen.

Table 11: EDX results of the particles found in the preheater after experiment STOWA 4.

Element	Massen%	Atom%
C	27.11	38.28
O	43.34	45.95
Na	0.40	0.29
Mg	3.75	2.62
Al	2.03	1.28
Si	3.34	2.01
P	10.10	5.53
S	0.20	0.11
K	2.99	1.30
Ca	4.84	2.05
Ti	0.29	0.10
Fe	1.60	0.49
Insgesamt	100.00	

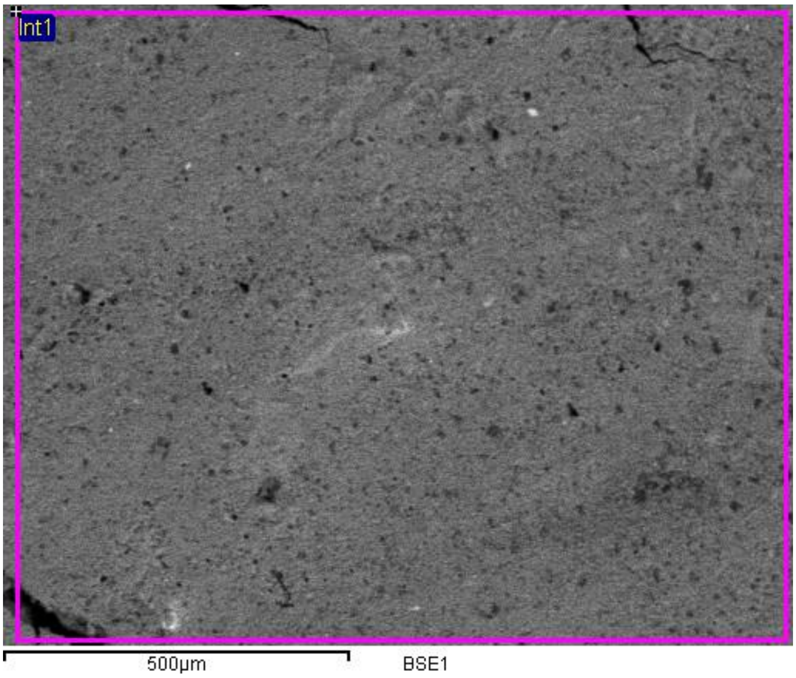
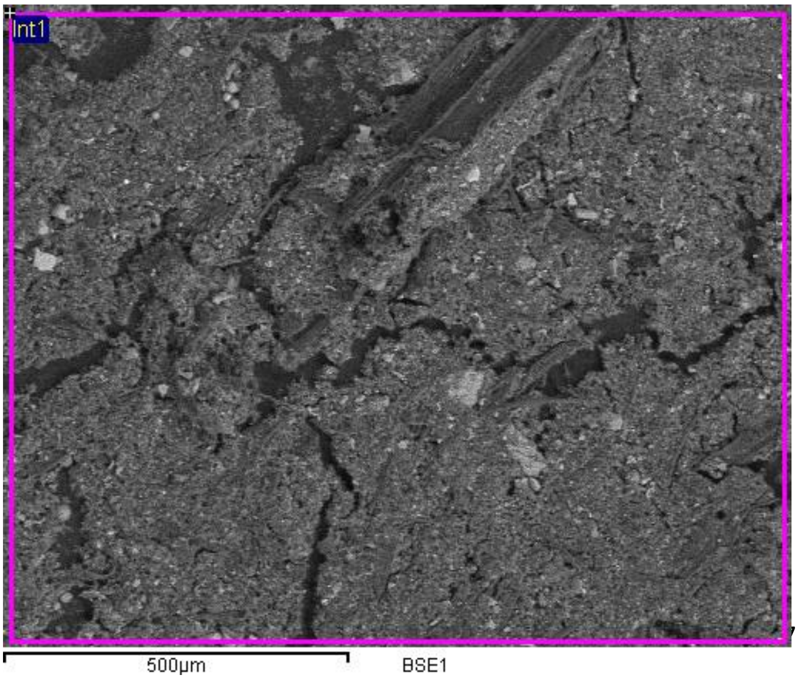


Table 12: EDX results of the particles found within the tee after experiment STOWA 4.

Element	Massen%	Atom%
C	53.50	63.90
N	0.00	0.00
O	34.15	30.62
Mg	1.18	0.70
Al	0.87	0.46
Si	2.19	1.12
P	3.39	1.57
S	0.44	0.20
K	1.16	0.43
Ca	1.95	0.70
Ti	0.22	0.06
Fe	0.79	0.20
Cu	0.15	0.03
Insgesamt	100.00	



Experiment conditions		
Dry matter content	15,88	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	650	°C
pressure	280	bar
real residence time	6,15	min
real Feed mass flow	186,0	g/h

Sewage sludge		
time	0,50	h
Total sludge fed	93,00	g
C- mass in the total feed*	6,306	g
Total mass effluent	33,0	g
Total mass salt birne	20,2	g
TOC-Conc.		
Effluent sample	2,96	mg/g
Salt conc. sample (Position 2)	8,19	mg/g
Gas production	9,69	Nl/h
Conversion of gasification (TC)	32,96	%
TOC-conversion**	90,79	%
Mass-balance	155,29	%
C-balance (TC)	46,49	%
C-deficit (TC)	-3,42	g
Gas composition		
H_2	29,18	Vol %
CO	0,47	Vol %
CO_2	31,18	Vol %
CH_4	31,09	Vol %
C_2H_4	0,15	Vol %
C_2H_6	7,89	Vol %
C_3H_6	0,01	Vol %
C_3H_8	0,03	Vol %
*TIC in the feed not detectable ** without the C from $KHCO_3$		

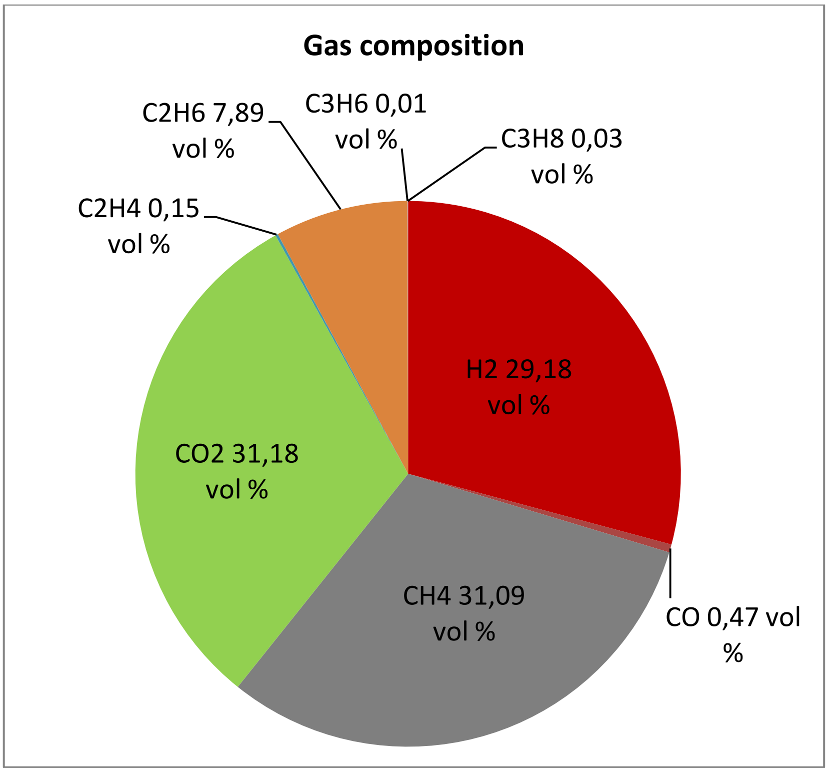
STOWA 4							
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**
Parameter	Concentration dry matter	Concentration diluted sludge[1]	after 1,5 hours	after 2 hours	after 3,5 hours	after experiment	after experiment
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]
TC	428	67966	1263	44	6624		
TIC	n.d.	n.d.	50	11	5272		
TOC (C)	428	67966	1213	33	1352		
H	60,9	9671	-	-	-		
N	68,6	10894	-	-	-		
O	-	-	-	-	-		
NO3	-	-	n.d.	n.d.	n.d.		
NO2	-	-	n.d.	n.d.	n.d.		
NH4	-	-	n.d.	108	8152		
TNb	-	-	417	4	5961		
P	29,6	4700	1,8	5,3	29,0		
PO4	-	-	n.d.	n.d.	n.d.		
S	7,6	1207	n.d.	n.d.	6,60		
SO4	-	-	n.d.	27,3	n.d.		
Ca	11,1	1763	4,0	6,8	2,8		
K	23,5[2]	4415	0,00	2,8	64,9		
Mg	6,4	1016	0,71	2,8	0,96		
Na	1,22	194	1,4	1,1	2,6		
Si	21,8	3462	16,8	16,5	28,2		
Al	4,68	743	0,13	0,09	0,20		
As	0,007	1,11	n.d.	n.d.	n.d.		
Cd	n.d.	n.d.	n.d.	n.d.	n.d.		
Cu	0,143	22,7	n.d.	n.d.	n.d.		
Pb	0,018	2,86	n.d.	n.d.	n.d.		
Zn	0,313	49,7	n.d.	n.d.	n.d.		
Cr	0,02	3,18	n.d.	n.d.	n.d.		
Fe	4,41	700	0,08	0,14	0,33		
Mo	0,006	0,95	n.d.	n.d.	n.d.		
Ni	0,013	2,06	n.d.	0,28	n.d.		
Cl	0,546	86,7	n.d.	n.d.	n.d.		
Hg	0,00049	0,0778	n.d.	n.d.	n.d.		
Phenolindex	-	-	-	-	-		

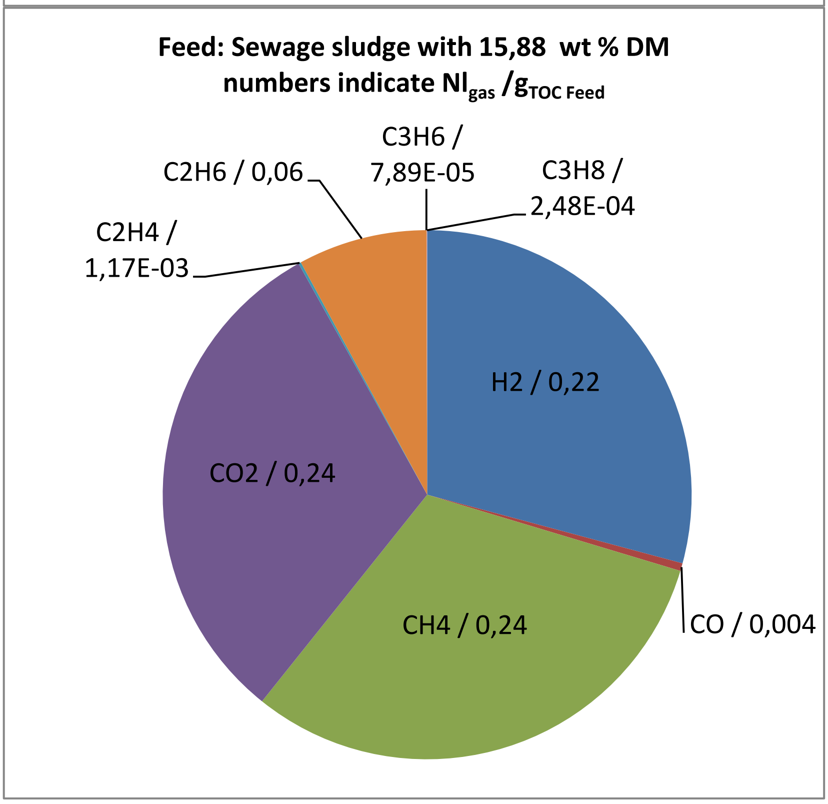
[1] for simplification it is assumed that the density of water equates to the density of sewage sludge
[2] calculated, because the charge 2 was only analysed once after addition of $KHCO_3$ for STOWA3

Legend:	n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Lelystad sample 21.3.14

Feed			
HHV:		18950	J/g
Ash content	by 550°C:	19,4	%
	by 815°C:	18,9	%
	by 1000°C:	18,6	%





2.4.5 Experiment STOWA 5

To avoid plugging the dry matter content was again lowered to 12,52 wt.-%. To investigate the influence of the residence time the feed flow rate was increased to obtain a residence time of 2,72 min. Because of a blockage of the flow rate during the feed phase the experiment STOWA 5 proceeded irregular and finally has to be stopped after 4,85 h. The plugging was localized in the sector of heater HZ 2. No detailed analysis was carried out. In Figure 7 the change in colour of the effluent samples of the feed phase is illustrated. An increasing brown coloration and turbidity over the reaction time can be observed. The third glass from the right contains even tarry dark brown suspended particles that stick to the glass wall.

Figure 7: Change in colour of the effluent samples during experiment STOWA 5

Experiment conditions		
Dry matter content	12,52	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	653	°C
pressure	280	bar
real residence time	2,72	min
real Feed mass flow	424,8	g/h

Sewage sludge		
time	2,52	h
Total sludge fed	1069	g
C- mass in the total feed*	57,2	g
Total mass effluent	804	g
Total mass salt birne	87,8	g
TOC-Conc.		
Effluent sample	2,96	mg/g
Salt conc. sample (Position 2)	0,32	mg/g
Gas production	28,8	Nl/h
Conversion of gasification (TC)	63,8	%
TOC-conversion**	94,9	%
Mass-balance	109,3	%
C-balance (TC)	80,9	%
C-deficit (TC)	11,05	g
Gas composition		
H_2	18,25	Vol %
CO	0,35	Vol %
CO_2	34,36	Vol %
CH_4	34,88	Vol %
C_2H_4	0,49	Vol %
C_2H_6	11,31	Vol %
C_3H_6	0,10	Vol %
C_3H_8	0,26	Vol %

*TIC in the feed not detectable
** without the C from $KHCO_3$

From experiment 5 until experiment 17 the masses of filter 1 and filter 2 were determined.

STOWA 5								**Filter solid**	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**Filter1 (Position 5)**	**Filter2 (Position 6)**
Parameter	Concentration dry matter	Concentration diluted sludge[1]	after 3,83 hours	after 4,3 hours	after 4,85 hours	after experiment	after experiment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	
TC	427	53460	10905	10512	10846	6797	4719	371	240
TIC	n.d.	n.d.	7460	7128	7428	4943	437	n.d.	n.d.
TOC (C)	427	53460	3444	3384	3418	1854	4281	371	240
H	60,9	7625	-	-	-	-	-	-	-
N	68,6	8588	-	-	-	-	-	28,3	n.d.
O	-	-	-	-	-	-	-	328	412
NO3	-	-	59	59	59	206	209	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NH4	-	-	13983	12660	12695	9115	2270	-	-
TNb	-	-	6100	7516	7497	5565	1506	-	-
P	29,6	3706	557	531	389	235	2674	70,1	111
PO4	-	-	1849	1778	1278	725	3415	-	-
S	7,6	952	89,0	112	342	340	324	6,4	2,5
SO4	-	-	138	138	101	70	700	-	-
Ca	11,1	1390	0,50	1,20	1,60	2,1	6,1	42,3	
K	29,8[2]	3481	909	920	681	420	1337	53,8	41,1
Mg	6,4	801	0,90	1,00	1,30	2,1	16	20,6	32,3
Na	1,22	153	17,3	17,8	12,1	9,1	98,1	2,8	3,7
Si	21,8	2729	35,4	35,2	35,5	44	81,9	35,7	40,6
Al	4,68	586	0,04	0,03	0,04	0,04	0,18	13,5	24,1
As	0,007	0,876	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,143	17,9	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.
Pb	0,018	2,25	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,313	39,2	n.d.	n.d.	n.d.	n.d.	2,62	n.d.	2,4
Cr	0,02	2,50	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Fe	4,41	552	n.d.	n.d.	n.d.	n.d.	0,74	18,6	23,3
Mo	0,006	0,751	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ni	0,013	1,63	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cl	0,546	68,4	23,7	23,6	23,4	27,7	45,5	n.d.	n.d.
Hg	0,00049	0,0613	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	473	517	512	352	518	-	-

[1] for simplification it is assumed that the density of water equates to the density of sewage sludge
[2] calculated, because the charge 2 was only analysed once after addition of $KHCO_3$ for STOWA3

Legend	
n.d.	not detectable or signal interference

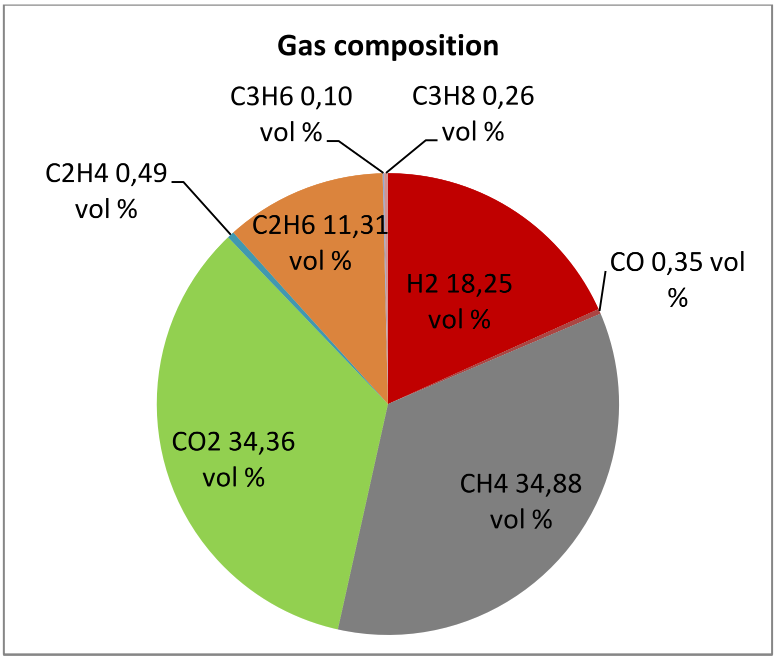
Additional analytical data: Sewage sludge from Lelystad sample 21.3.14

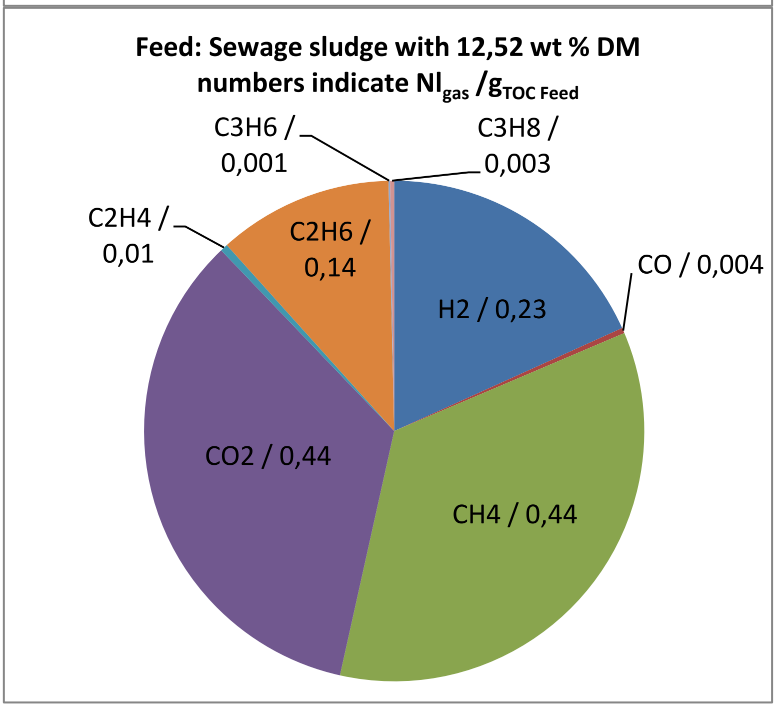
Feed			
HHV:		18950	J/g
Ash content:	by 550°C:	19,4	%
	by 815°C:	18,9	%
	by 1000°C:	18,6	%

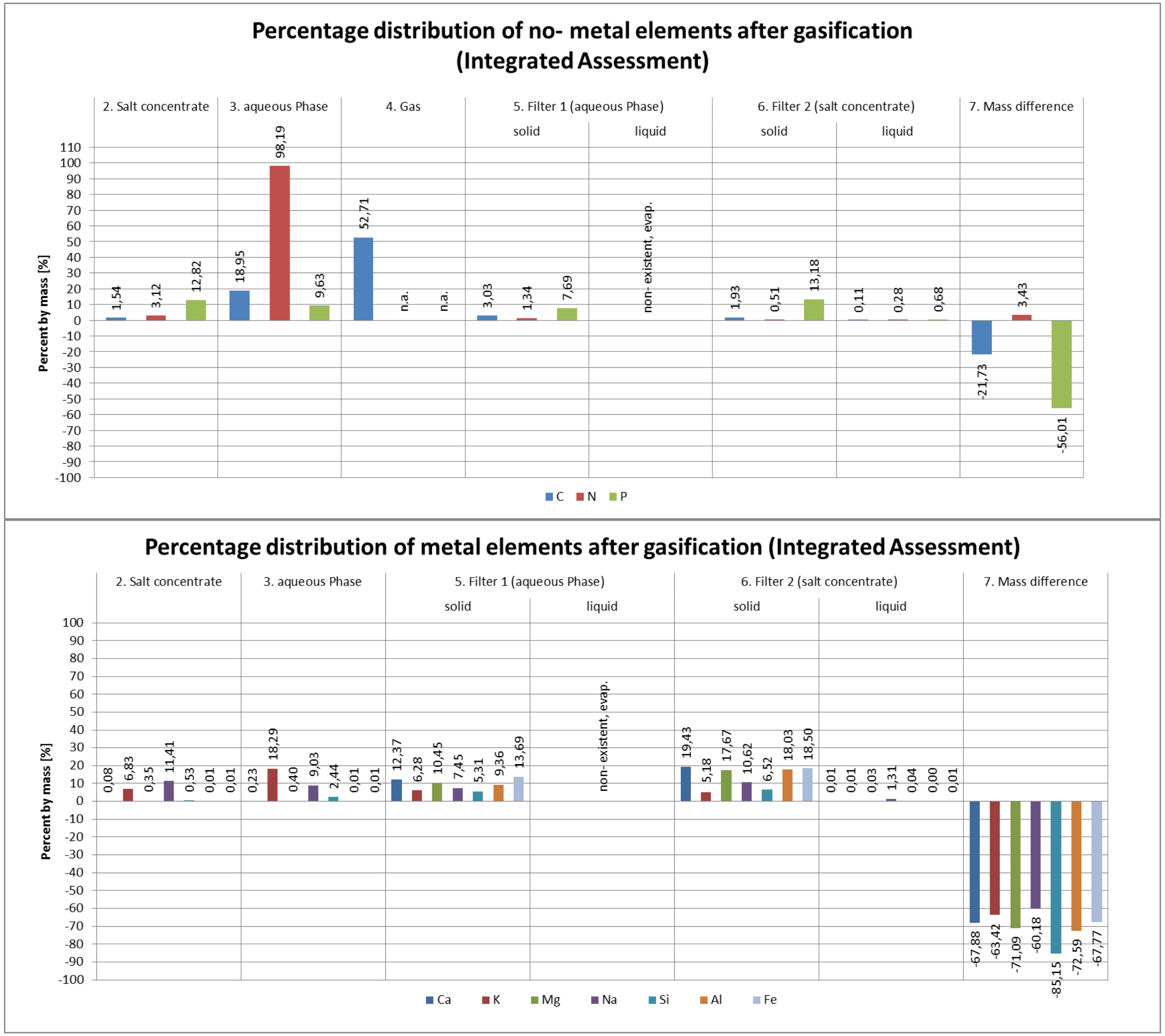
Mass of solids:

filter1 - aqueous Phase	m=	11,4	g
filter2 - salt concentrate	m=	12,3	g

Method: EDX (except N)







The cleaning phase with cold water is not incorporated in the total balance of experiment STOWA 5.

2.4.6 Experiment STOWA 6

Experiment STOWA 6 failed. After the beginning of the feed phase the pressure increased immediately. After 0,75 h the test rig was plugged and the experiment had to be stopped. Due to lack of data the experiment cannot be evaluated.

2.4.7 Experiment STOWA 7

In experiment STOWA 7 the temperature of the preheater was increased to 480 °C in order to optimize the salt separation. The experiment ran stable within the feed phase. During the cleaning phase with cold water a blockage of the flow rate in the preheater in a distance of 53 – 55 cm from the entrance occurred. Furthermore the outlet line of the reactor 15 cm from the end of the reactor was also plugged. The compositions of the particles from both positions that were blocked were analysed by EDX (see Table 13 and Table 14).

Table 13: EDX results of the greyish black particles found in the preheater after STOWA 7.

Element	Mass%	Atom%
C	25.09	36.58
O	41.32	45.23
Na	0.29	0.22
Mg	2.02	1.45
Al	2.19	1.42
Si	7.72	4.81
P	8.72	4.93
S	0.14	0.07
K	4.05	1.81
Ca	6.37	2.79
Ti	0.42	0.15
Cr	0.17	0.06
Fe	1.26	0.39
Ni	0.25	0.07
total	100.00	

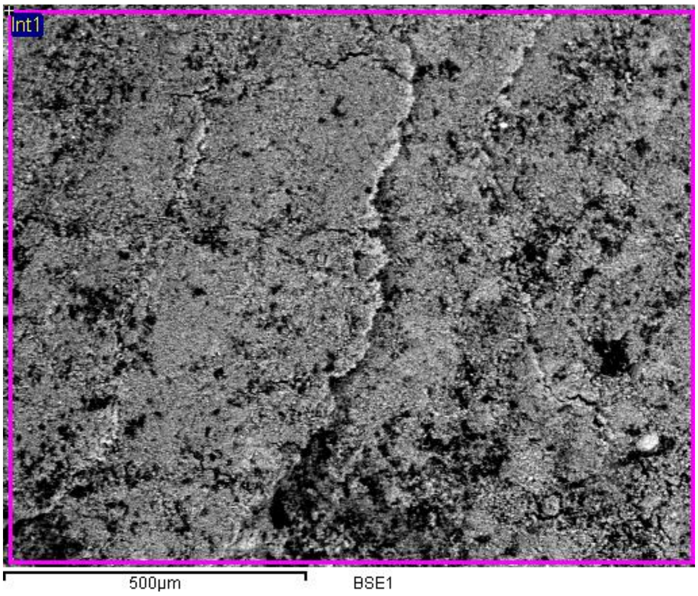
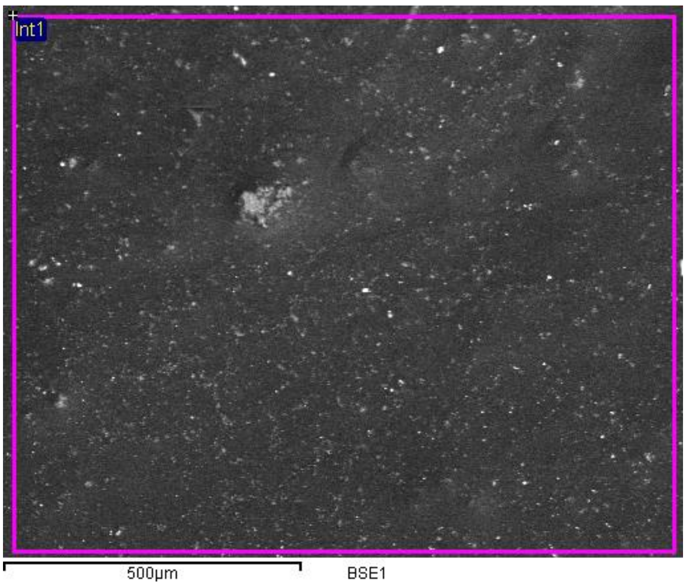


Table 14: EDX results of the pitch-black particles found in the outlet line of the reactor after STOWA 7.

Element	Mass %	Atom%
C	92.70	96.41
O	2.57	2.01
Mg	0.11	0.06
Al	0.14	0.07
Si	0.67	0.30
P	0.59	0.24
S	0.76	0.30
K	0.55	0.18
Ca	0.42	0.13
Fe	0.33	0.07
Ni	1.14	0.24
total	100.00	



Both samples mainly consist of carbon and oxygen. However the tarry material of the outlet line of the reactor mainly consists only of carbon. An additional GC-MS-analysis showed that the major constituents of the tarry sample are phenanthrenes, pyrenes, indoles and fluorenes. After the feed phase the gasification of residues in the reactor proceeds. However the gasification yield cannot be calculated because the amount of dry matter within the reactor is not known and no more carbon is fed. The gas from the residual gasification that leaves the plant still contains carbon containing components. Via GC-MS-analysis and comparison with the database NIST 05 mass spectral library a trace analysis was carried out. Thereby mainly benzene, toluene, m,p-xylene, ethylbenzene and o-xylene was detected. This is an indication for tar formation during the process.

Experiment conditions		
Dry matter content	12,33	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	652	°C
pressure	280	bar
real residence time	2,78	min
real Feed mass flow	417,5	g/h

Sewage sludge		
Time	4,0	h
Total sludge fed	1670	g
C- mass in the total feed*	88,95	g
Total mass effluent	1228	g
Total mass salt birne	62,0	g
TOC-Conc.		
Effluent sample	1,61	mg/g
Salt conc. sample (Position 2)	0,15	mg/g
Gas production	28,23	Nl/h
Conversion of gasification (TC)	64,0	%
TOC-conversion**	97,3	%
Mass-balance	105,9	%
C-balance (TC)	77,9	%
C-deficit (TC)	-20,0	g
Gas composition		
H_2	18,52	Vol %
CO	0,05	Vol %
CO_2	35,82	Vol %
CH_4	32,99	Vol %
C_2H_4	0,42	Vol %
C_2H_6	11,86	Vol %
C_3H_6	0,08	Vol %
C_3H_8	0,27	Vol %

*TIC in feed not detectable

** without the C from $KHCO_3$

STOWA 7									Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**solid pieces in the preheater**	**Filter1 (Position 5)**	**Filter2 (Position 6)**
Parameter	Concen-tration dry matter	Concentration diluted sludge*	after 4,5 hours	after 5,5 hours	after 6,5 hours	after experi-ment	after experiment	grey, dark pieces	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[weight %]	[mg/g]	
TC	433	53389	7934	8337	7806	5043	1309	25,1	751	250
TIC	n.d.	n.d.	6240	6507	6153	3809	191	-	n.d.	n.d.
TOC (C)	433	53389	1694	1830	1653	1234	1118	-	751	250
H	62,9	7756	-	-	-	-	-	-	-	-
N	73	9001	-	-	-	-	-	n.d.	n.d.	13,3
O	-	-	-	-	-	-	-	41,3	137	408
NO3	-	-	207	207	208	n.d.	32,6	-	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NH4	-	-	13124	12397	13514	8294	374	-	-	-
TNb	-	-	6921	7513	7294	4917	384	-	-	-
P	30,8	3798	218	220	226	117	860	8,72	17,9	115
PO4	-	-	561	574	553	312	2061	-	-	-
S	7,32	903	1865	1615	1437	115	174	0,14	6,1	2,7
SO4	-	-	52,2	54,2	50,3	39,4	330	-	-	-
Ca	12,7	1566	0,50	0,67	0,89	1,85	4,85	6,37	12,3	57,3
K	11,8	1455	515	525	596	305	2125	4,05	13,2	52,7
Mg	7,02	866	0,48	1,60	2,80	5,5	35,5	2,02	5,8	26,3
Na	1,25	154	7,40	7,50	5,20	6,1	64,1	0,29	0,5	3,3
Si	21,6	2663	47,0	48,0	50,0	38	42,5	7,72	17,8	38,9
Al	5,21	642	0,14	0,12	0,12	0,22	0,4	2,19	5,7	24,9
As	0,007	0,863	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,153	18,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Pb	0,012	1,48	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,325	40,1	0,01	0,01	0,02	0,02	0,12	-	n.d.	n.d.
Cr	0,018	2,22	n.d.	n.d.	n.d.	n.d.	n.d.	0,17	2,8	n.d.
Fe	3,87	477	n.d.	n.d.	n.d.	n.d.	2,20	1,26	6,8	17,1
Mo	0,006	0,740	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ti	-	-	-	-	-	-	-	0,42	n.d.	n.d.
Ni	0,012	1,48	n.d.	n.d.	n.d.	n.d.	108	0,25	21,8	n.d.
Cl	0,67	82,6	27,6	27,8	27,6	27,6	43,9	n.d.	n.d.	n.d.
Hg	0,00055	0,0678	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	86	337	325	200	25	-	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

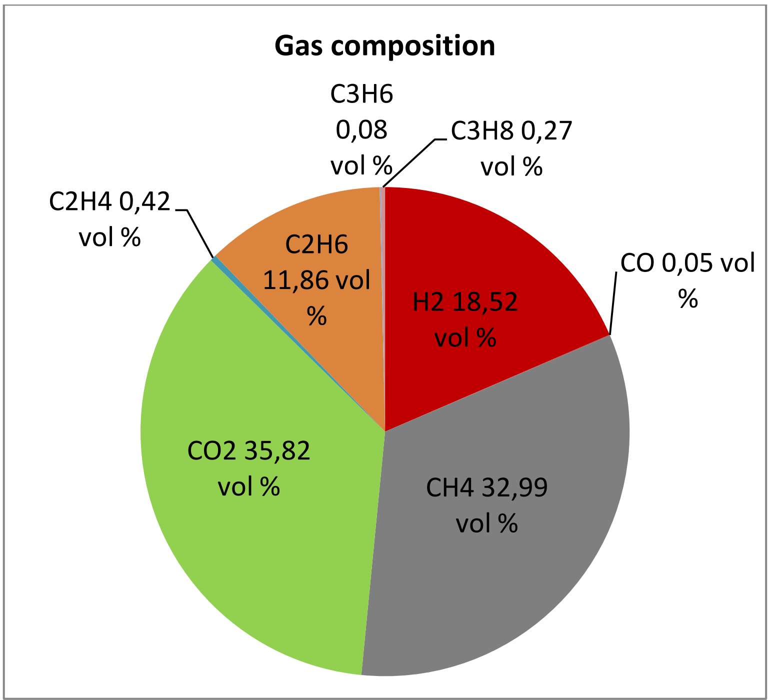
Additional analytical data: Sewage sludge from Lelystad sample 29.4.14

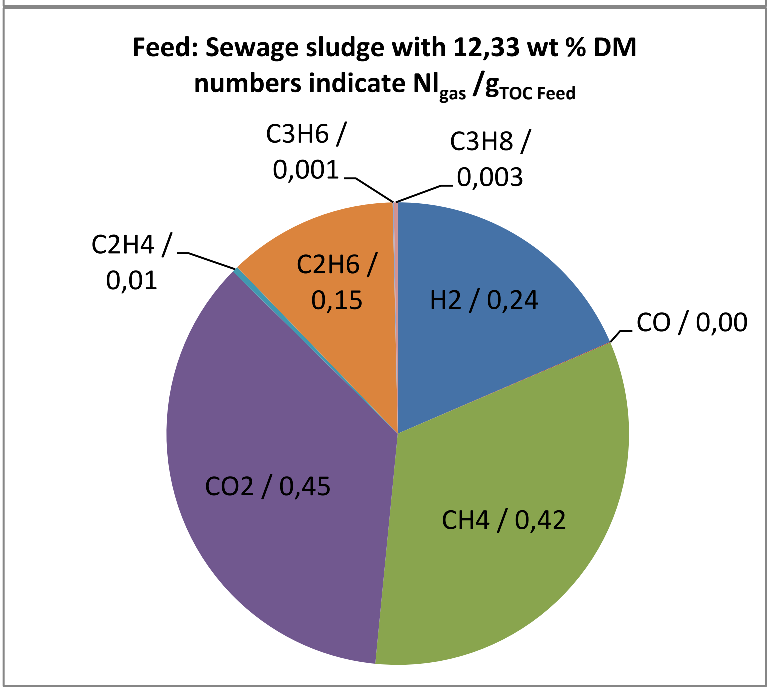
Feed			
HHV:		18601	J/g
Ash content	by 550°C:	20	%
	by 815°C:	18,7	%
	by 1000°C:	18,6	%

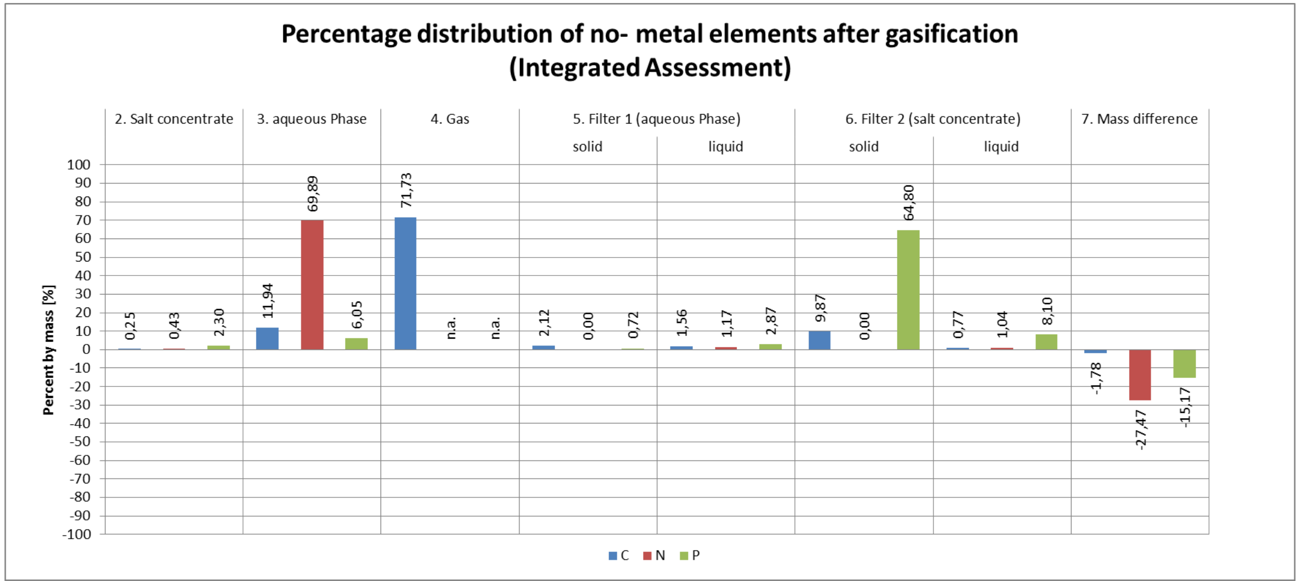
Mass of solids

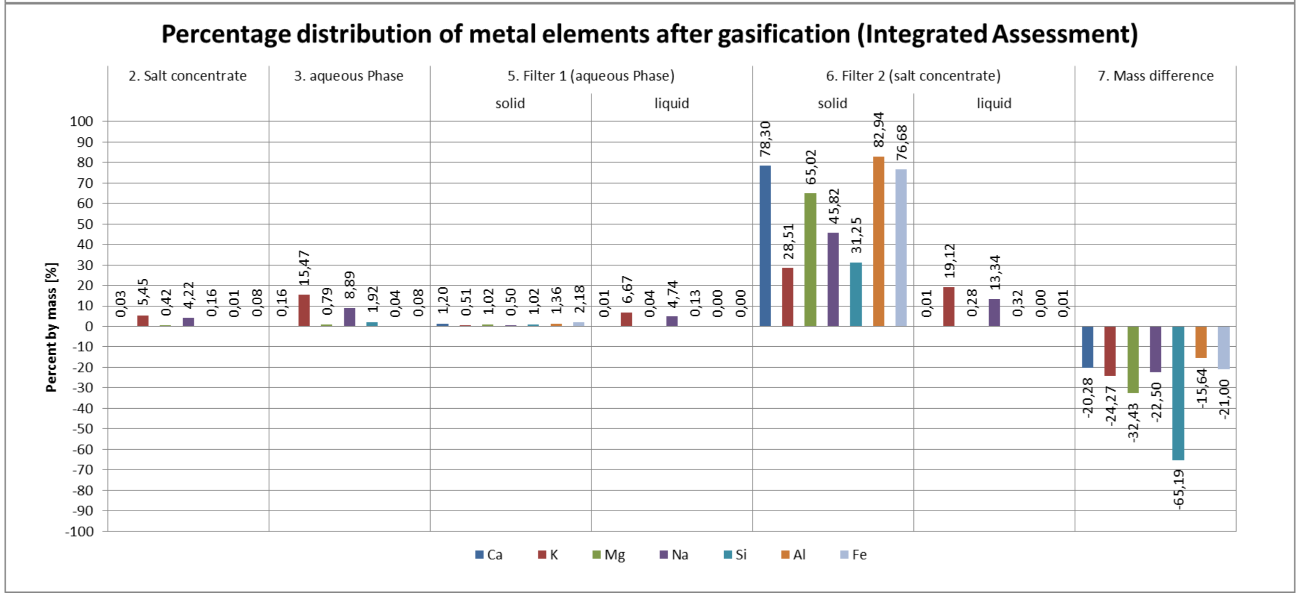
filter1 - aqueous Phase	m=	4,53	g
filter2 - salt concentrate	m=	63,4	g

Method: EDX (except N-determination)









The cleaning phase with cold water is not incorporated in the total balance of experiment STOWA 7.

2.4.8 Experiment STOWA 8

The reaction temperature was lowered to 619 °C in comparison to the experiments STOWA 3 – STOWA 7 where a reaction temperature of about 650 °C was set. This experiment should show whether a satisfactory gasification yield is achievable after the other parameters were optimized. Experiment STOWA 8 ran stable.

Experiment conditions		
Dry matter content	12,62	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	619	°C
pressure	280	bar
real residence time	2,76	min
real Feed mass flow	412,0	g/h

Sewage sludge		
time	3,00	h
Total sludge fed	1236	g
C- mass in the total feed*	67,7	g
Total mass effluent	801	g
Total mass salt birne	86,1	g
TOC-Conc.		
Effluent sample	3,07	mg/g
Salt conc. sample (Position 2)	12,76	mg/g
Gas production	23,35	Nl/h
Conversion of gasification (TC)	49,9	%
TOC-conversion**	93,4	%
Mass-balance	94,9	%
C-balance (TC)	65,8	%
C-deficit (TC)	23,5	g
Gas composition		
H_2	22,01	Vol %
CO	0,46	Vol %
CO_2	40,89	Vol %
CH_4	25,41	Vol %
C_2H_4	0,87	Vol %
C_2H_6	8,28	Vol %
C_3H_6	0,55	Vol %
C_3H_8	1,52	Vol %

*TIC in the feed not detectable
** without the C from $KHCO_3$

STOWA 8									Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**analyses filter 1 (tar)**	**Filter1 (Position 5)**	**Filter2 (Position 6)**
Parameter	Concentration dry matter	Concentration diluted sludge*	after 4,,83 hours	after 5,83 hours	after 6,83 hours	after experiment	after experiment	Concentration	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	[mg/g]	
TC	434	54771	9929	9777	9101	6399	11423	922	869	252
TIC	n.d.	n.d.	6822	6739	6031	3897	203	-	-	-
TOC (C)	434	54771	3107	3038	3070	2502	11220	-	-	-
H	63,7	8039	-	-	-	-	-	-	-	-
N	72,8	9188	-	-	-	-	-	n.d.	n.d.	n.d.
O	-	-	-	-	-	-	-	60,2	90,8	407
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NH4	-	-	12600	10900	11900	7678	624	-	-	-
TNb	-	-	6755	6408	6576	4639	903	-	-	-
P	31,6	3988	72,0	647	73,2	54,8	1505	3,2	8,3	113,6
PO4	-	-	187	162	182	150	4033	-	-	-
S	11,1	1401	111	176	120	93	332	3,6	4,4	4,5
SO4	-	-	70,8	60,8	63,9	59	682	-	-	-
Ca	14	1767	0,98	1,28	0,85	2,25	4,55	1,7	5,3	51,6
K	11,4	1439	185	191	223	154	3990	2,8	5,4	66,4
Mg	7,2	909	1,11	1,06	1,42	2,05	10,4	0,8	1,4	26,7
Na	1,05	133	3,55	4,27	4,25	3,48	112	n.d.	n.d.	2,4
Si	26,3	3319	58,5	53,8	53,0	45,5	76,8	0,41	n.d.	35,4
Al	5,6	707	0,13	0,09	0,11	0,11	0,29	0,13	3,4	21,9
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,18	22,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Pb	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,32	40,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cr	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,7
Fe	3,8	480	n.d.	n.d.	n.d.	0,9	0,29	n.d.	2	14,4
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ni	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1	n.d.
Cl	0,572	72,2	27,1	26,8	26,8	27,6	65,7	n.d.	n.d.	n.d.
Hg	0,0003	0,0379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	511	533	537	390	38	-	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Lelystad sample 20.5.14

Feed			
HHV:		19161	J/g
Ash content	by 550°C:	18,4	%
	by 815°C:	16,6	%
	by 1000°C:	16,5	%

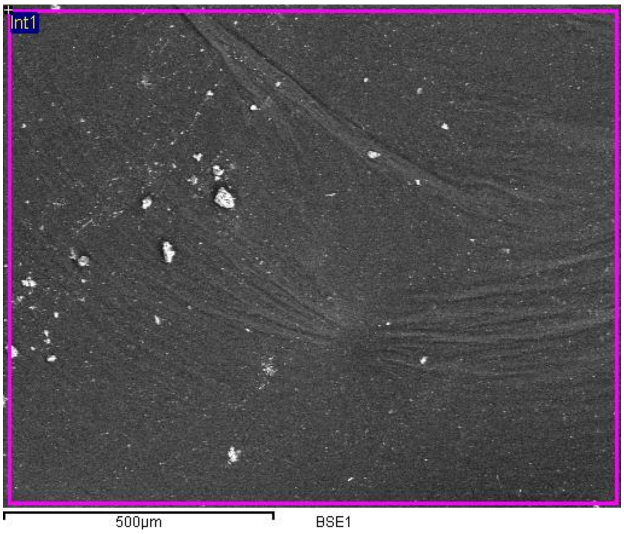
Mass of solids:

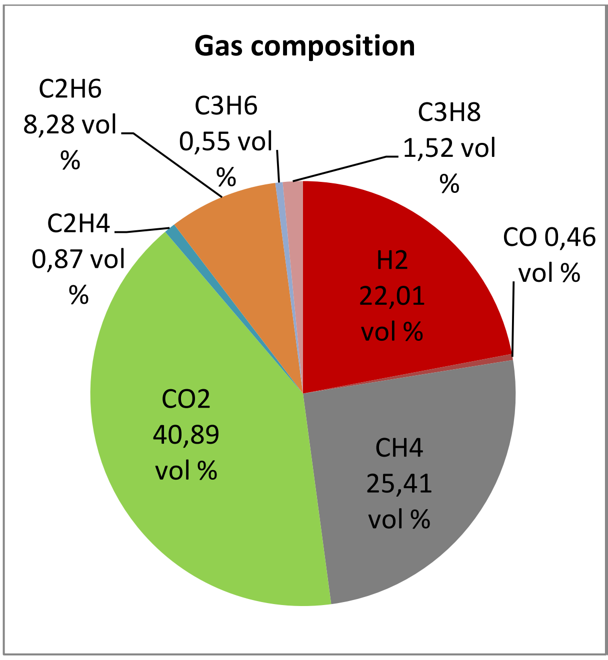
filter1 tar	m=	1,05 g
filter1 - aqueous Phase	m=	4,83 g
filter2 - salt concentrate	m=	64,3 g
Methode:	EDX all values	

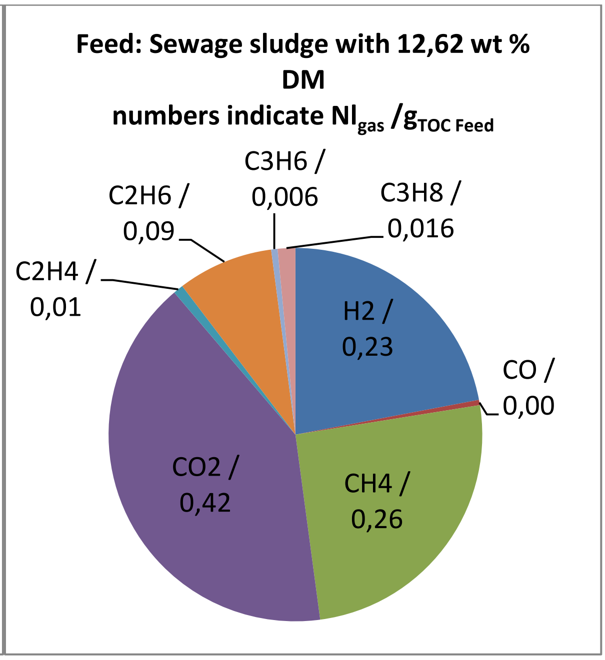
The composition of the tarry fraction of filter F01 was analysed by EDX. The results are listed in Table 15. The analysis shows that the particles mainly consist of carbon.

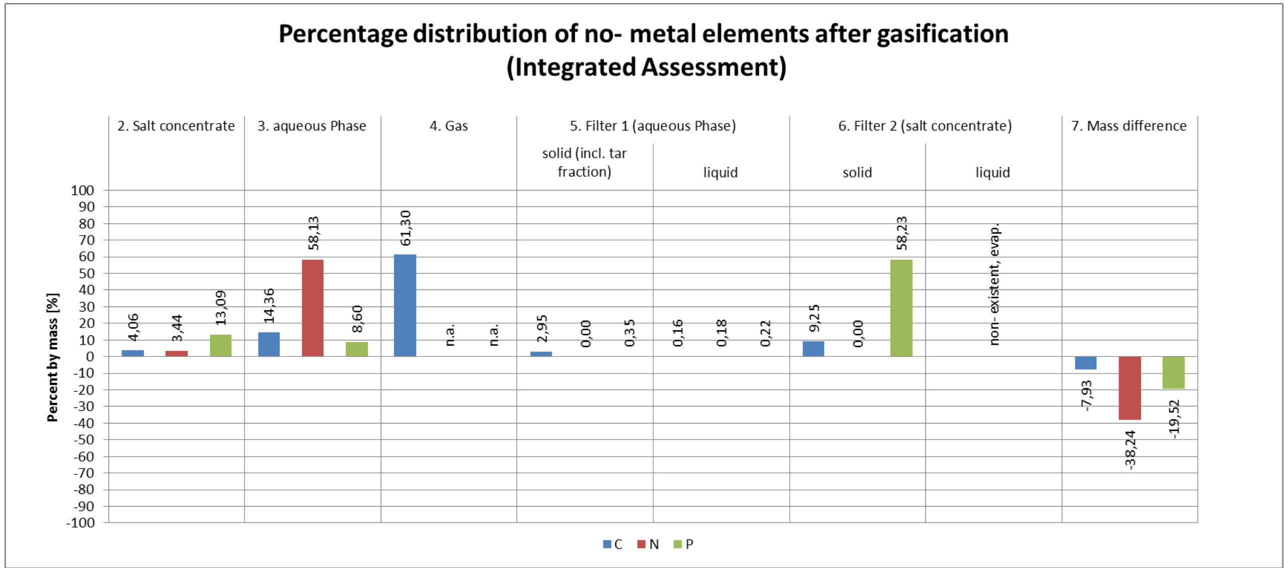
Table 15: EDX results of the tarry fraction of filter of STOWA 8.

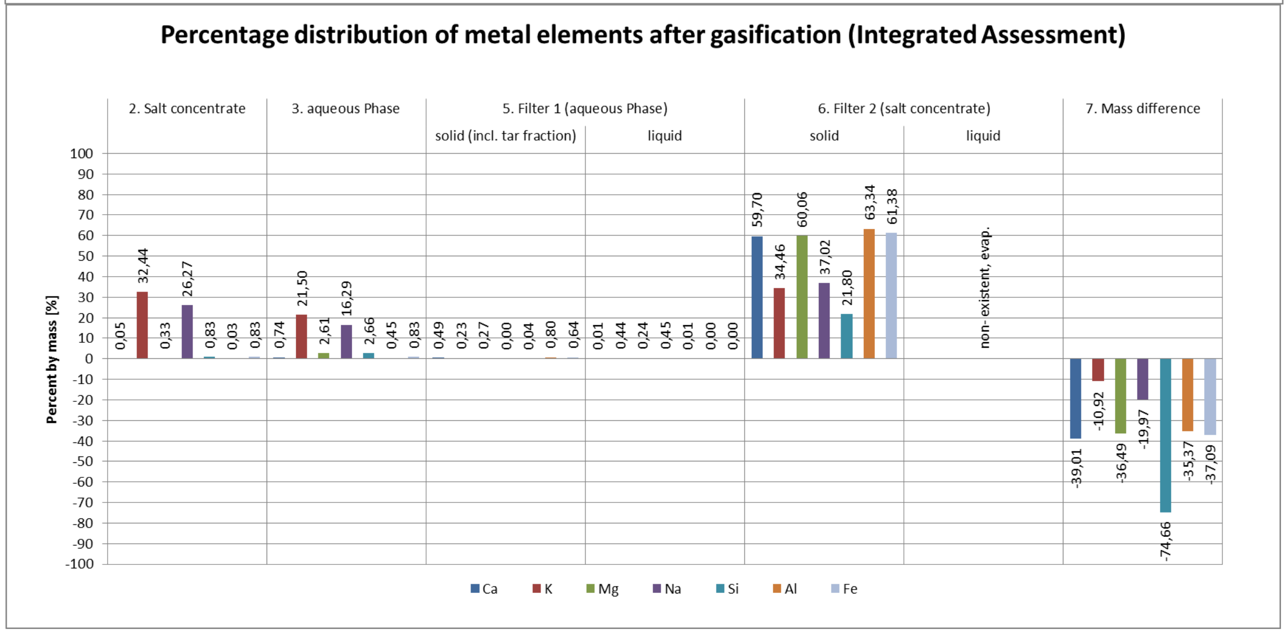
Element	Mass%	Atom%
C	92.23	94.67
O	6.02	4.64
Mg	0.08	0.04
Al	0.13	0.06
Si	0.41	0.18
P	0.32	0.13
S	0.36	0.14
K	0.28	0.09
Ca	0.17	0.05
total	100.00	








Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
solid (incl. tar fraction)
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
4,06
3,44
13,09
14,36
58,13
8,60
61,30
n.a.
n.a.
2,95
0,00
0,35
0,16
0,18
0,22
9,25
0,00
58,23
non- existent, evap.
-7,93
-38,24
-19,52
C
N
P


Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
solid (incl. tar fraction)
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
0,05
32,44
0,33
26,27
0,83
0,03
0,83
0,74
21,50
2,61
16,29
2,66
0,45
0,83
0,49
0,23
0,27
0,00
0,04
0,80
0,64
0,01
0,44
0,24
0,45
0,01
0,00
0,00
59,70
34,46
60,06
37,02
21,80
63,34
61,38
non- existent, evap.
-39,01
-10,92
-36,49
-19,97
-74,66
-35,37
-37,09
Ca
K
Mg
Na
Si
Al
Fe

2.4.9 Experiment STOWA 9

For experiment STOWA 9, it was intended not to use salt separation at all. But the salt separation system had to be used due to pressure increase in the system – an indication of beginning of clogging. By this measure the experiment STOWA 9 ran stable. For an experiment without salt separation other hardware should be used.

Experiment conditions		
Dry matter content	11,69	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	653	°C
pressure	280	bar
real residence time	2,75	min
real Feed mass flow	423,0	g/h

Sewage sludge		
time	5,00	h
Total sludge fed	2115	g
C- mass in the total feed*	107,3	g
Total mass effluent	1512	g
Total mass salt brine	150	g
TOC-Conc.		
Effluent sample	0,98	mg/g
Salt conc. sample (Position 2)	3,25	mg/g
Gas production	30,14	Nl/h
Conversion of gasification (TC)	67,9	%
TOC-conversion**	97,8	%
Mass-balance	103,4	%
C-balance (TC)	80,7	%
C-deficit (TC)	-21,1	g
Gas composition		
H_2	21,81	Vol %
CO	0,04	Vol %
CO_2	34,25	Vol %
CH_4	31,80	Vol %
C_2H_4	0,38	Vol %
C_2H_6	11,27	Vol %
C_3H_6	0,10	Vol %
C_3H_8	0,36	Vol %
*TIC in the feed not detectable		
** without the C from $KHCO_3$		

STOWA 9								Filter solid	
	Feed analysis		aq. Effluent analysis during experiment (Position 3)				salt concentrate analysis (Position 2)	Filter1 (Position 5)	Filter2 (Position 6)
Parameter	Concentration dry matter	Concentration diluted sludge*	after 4,5 hours	after 5,5 hours	after 6,5 hours	after experi-ment	after experiment *	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	
TC	434	50735	9750	10400	9890	7250	3385	690	220
TIC	n.d.	n.d.	7800	8420	7960	5700	135	-	-
TOC (C)	434	50735	1950	1980	1930	1550	3250	-	-
H	63,7	7447	-	-	-	-	-	-	-
N	72,8	8510	-	-	-	-	-	n.d.	n.d.
O	-	-	-	-	-	-	-	165	435
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NH4	-	-	11840	11902	11431	8097	79	-	-
TNb	-	-	9400	9200	9300	6200	821	-	-
P	31,6	3694	172	203	295	251	278	34,6	122
PO4	-	-	394	475	640	563	746	-	-
S	11,1	1298	108	439	1093	537	30,0	6,2	2,1
SO4	-	-	77,8	84,2	92,1	82,4	67,6	-	-
Ca	14	1637	0,78	0,68	0,83	1,76	4,72	25,9	64,6
K	11,4	1333	523	597	744	590	545	21,5	42,7
Mg	7,2	842	3,76	4,28	19,2	14,8	61,2	7,1	28,8
Na	1,05	123	7,51	8,07	10,7	11,0	16,5	1,3	2,6
Si	26,3	3075	57,0	58,6	59,5	59,4	14,0	26,4	34,3
Al	5,6	655	0,14	n.d.	n.d.	0,16	0,17	10,8	27,5
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,18	21,0	n.d.	n.d.	n.d.	n.d.	n.d.	1	n.d.
Pb	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,32	37,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cr	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Fe	3,8	444	n.d.	n.d.	n.d.	0,04	1,31	7,7	17,3
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2,02	n.d.	n.d.
Ni	n.d.	n.d.	n.d.	6,40	n.d.	n.d.	n.d.	1,2	n.d.
Cl	0,572	66,9	27,3	27,3	27,7	27,9	30,1	n.d.	n.d.
Hg	0,0003	0,035	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	331	334	345	276	7	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

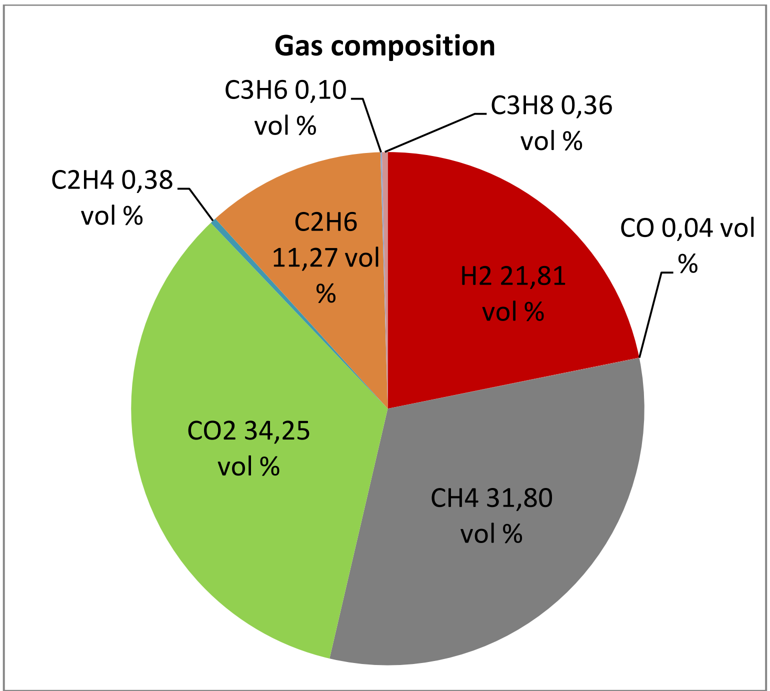
Additional analytical data: Sewage sludge from Lelystad sample 20.5.14

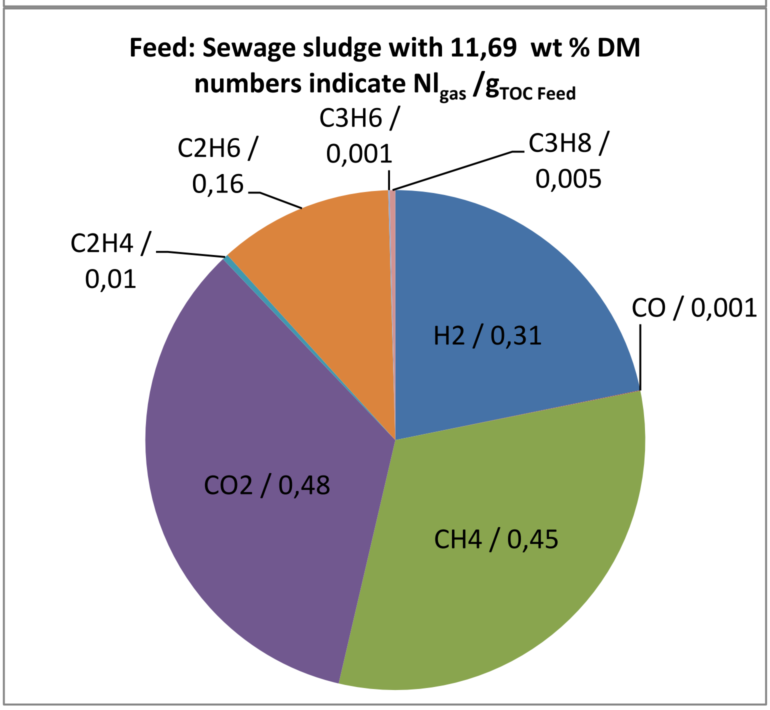
Feed			
HHV:		19161	J/g
Ash content	by 550°C:	18,4	%
	by 815°C:	16,6	%
	by 1000°C:	16,5	%

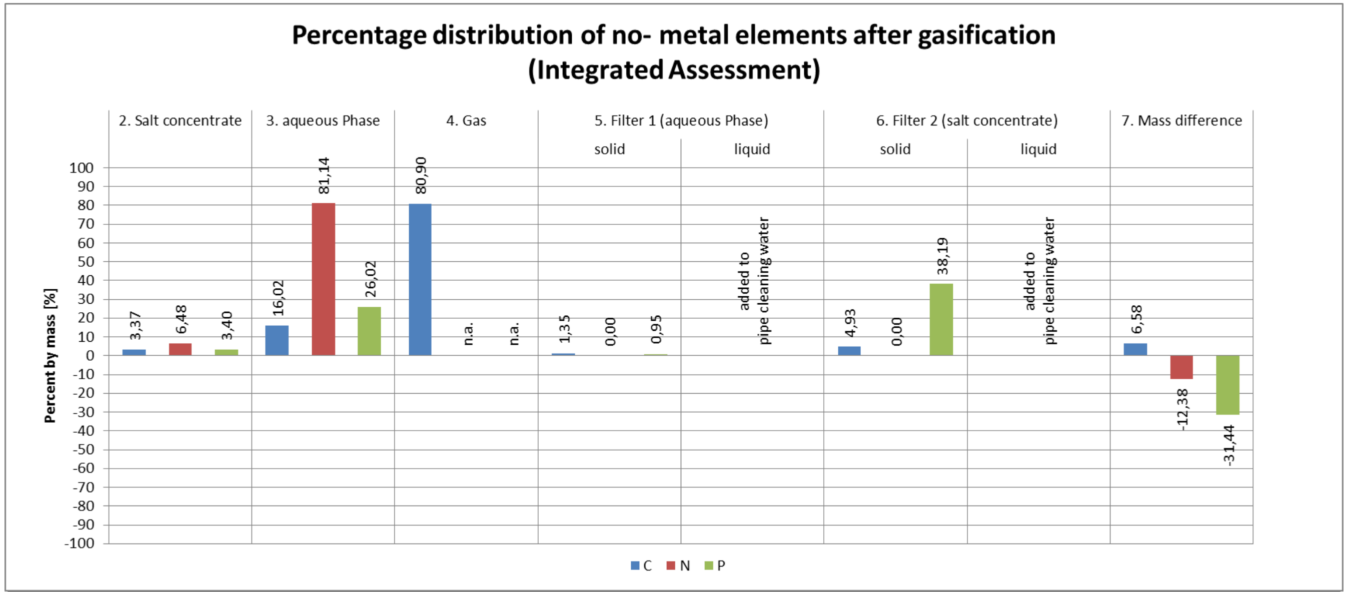
Mass of solids:

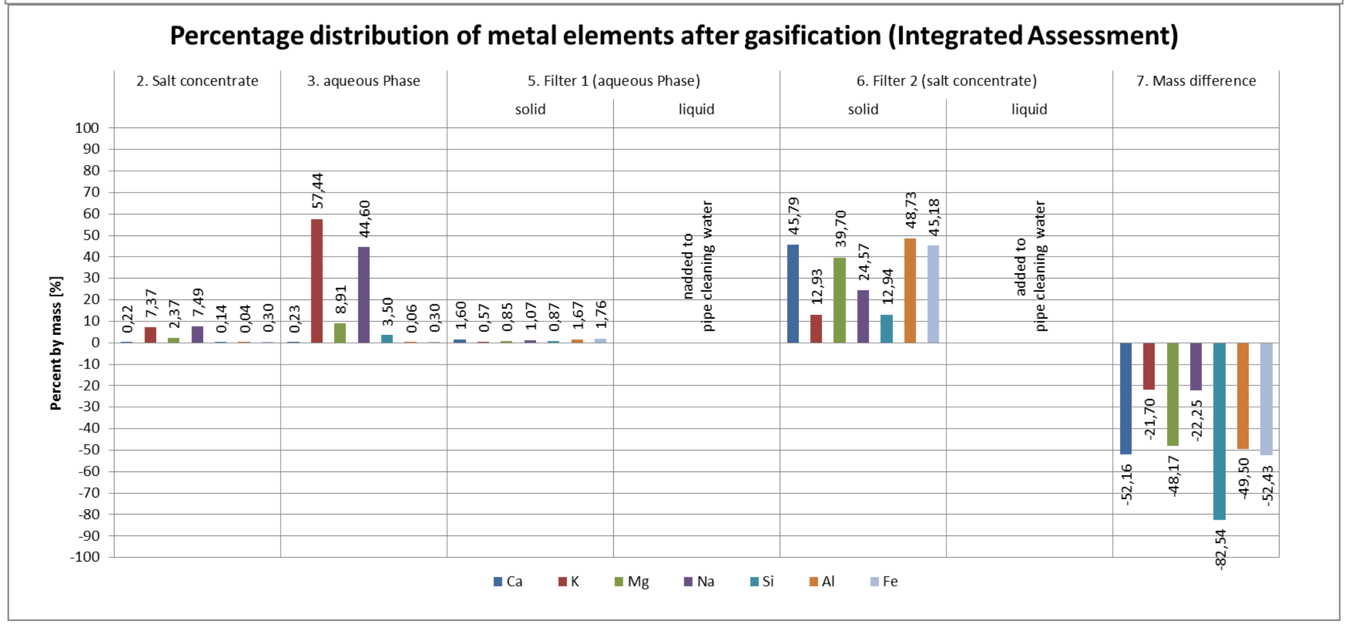
Filter1 – aqueous phase m = 3,1 g
Filter 2 – salt concentrate m = 35,5 g

Method: EDX (all values)






Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
3,37
6,48
3,40
16,02
81,14
26,02
80,90
n.a.
n.a.
1,35
0,00
0,95
added to pipe cleaning water
4,93
0,00
38,19
added to pipe cleaning water
6,58
-12,38
-31,44
C
N
P


Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,22
7,37
2,37
7,49
0,14
0,04
0,30
0,23
57,44
8,91
44,60
3,50
0,06
0,30
1,60
0,57
0,85
1,07
0,87
1,67
1,76
nadded to pipe cleaning water
45,79
12,93
39,70
24,57
12,94
48,73
45,18
added to pipe cleaning water
-52,16
-21,70
-48,17
-22,25
-82,54
-49,50
-52,43
Ca
K
Mg
Na
Si
Al
Fe

2.4.10 Experiment STOWA 10

The first experiment with sewage sludge from Oijen, STOWA 10 ran stable. Only short time before the end of the feed phase a blocking of the feed flow began. As a result the cleaning phase proceeded irregularly.

Experiment conditions		
Dry matter content	12,71	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	649	°C
pressure	280	bar
real residence time	2,76	min
real Feed mass flow	421,1	g/h

Sewage sludge		
time	3,93	h
Total sludge fed	1656,50	g
C-mass in the total feed*	87,37	g
Total mass effluent	1119,31	g
Total mass salt brine	96,09	g
TOC-Conc.		
Effluent sample	2,11	mg/g
Salt conc. sample (Position 2)	0,33	mg/g
Gas production	30,59	Nl/h
Conversion of gasification (TC)	71,2	%
TOC-conversion**	96,7	%
Mass-balance	96,9	%
C-balance (TC)	81,8	%
C-deficit (TC)	-16,2	g
Gas composition		
H_2	17,18	Vol %
CO	0,22	Vol %
CO_2	33,36	Vol %
CH_4	35,08	Vol %
C_2H_4	0,61	Vol %
C_2H_6	12,90	Vol %
C_3H_6	0,17	Vol %
C_3H_8	0,47	Vol %
* TIC in the feed not detectable		
** without the C from $KHCO_3$		

STOWA10									Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**in the effluent glass jar**	**Filter1 (Position 5)**	**Filter2 (Position 6)**
Parameter	Concentra-tion dry matter	Concentration diluted sludge*	after 4 hours	after 5 hours	after 6 hours	after experi-ment	after experiment	dark sediment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	weight [%]	[mg/g]	
TC	415	52747	7712	7759	7437	5590	2606	29,5	249	219 *(336)*
TIC	n.d.	n.d.	5537	5637	5309	4002	137	-	n.d.	n.d.
TOC (C)	415	52747	2175	2121	2128	1589	2468	-	249	219 *(336)*
H	60,5	7690	-	-	-	-	-	-	-	-
N	42,2	5364	-	-	-	-	-	n.d.	12,9	11,8
O	-	-	-	-	-	-	-	37	379	392
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NH4	-	-	8637	9332	8494	6412	352	-	-	-
TNb	-	-	5601	5566	5572	4181	604	-	-	-
P	24	3050	272	300	341	219	265	9,49	75,9	76,3 *(74,6)*
PO4	-	-	640	788	857	513	517	-	-	-
S	7,8	991	512	657			187	1,26	13,8	n.d.
SO4	-	-	174	176	180	113	82,8	-	-	-
Ca	22,5	2860	10,8	16,2	18,6	16,8	14,9	1,53	81,1	70,8
K	3,6	458	1487	1469	1576	1110	414	10,7	45	27,5
Mg	3,1	394	5,65	5,75	3,05	4,45	147	4,05	8,7	10,5
Na	0,88	112	12,0	20,5	23,6	14,3	13,1	0,22	2,9	2
Si	26,5	3368	78,0	78,6	78,6	719	32,8	2,32	100	109
Al	12,2	1551	0,10	0,17	0,12	0,2	1,09	0,8	44,7	38,2
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	5,6	712	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2	1,77
Pb	0,11	14	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,5	0,4
Zn	0,86	109	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	3,3	2,8
Cr	0,039	4,96	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,59
Fe	13,8	1754	0,50	0,53	0,09	0,13	16,40	1,82	47,9	43,8
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,34	n.d.	0,06	0,09
Ni	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cl	1,91	243	54,2	58,1	55,2	54,5	35,1	1,18	0,317	0,067
Mn	-	-	-	-	-	-	-	0,07	0,57	0,62
Hg	0,0003	0,0381	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-		502	503	518	378	30	-	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

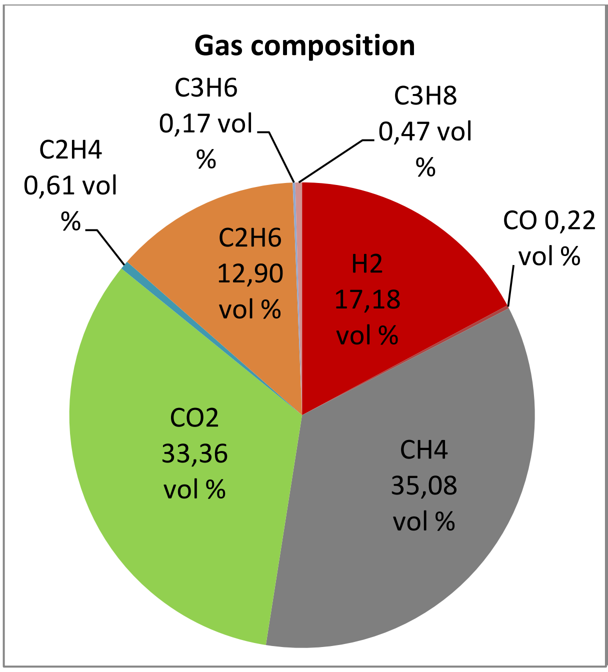
Legend	
n.d.	not detectable or signal interference

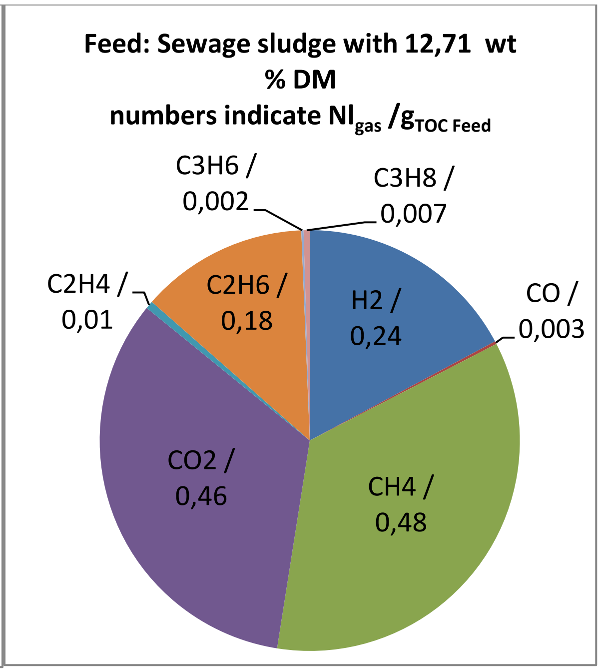
Additional analytical data: Sewage sludge from Oijen sample 11.06.14

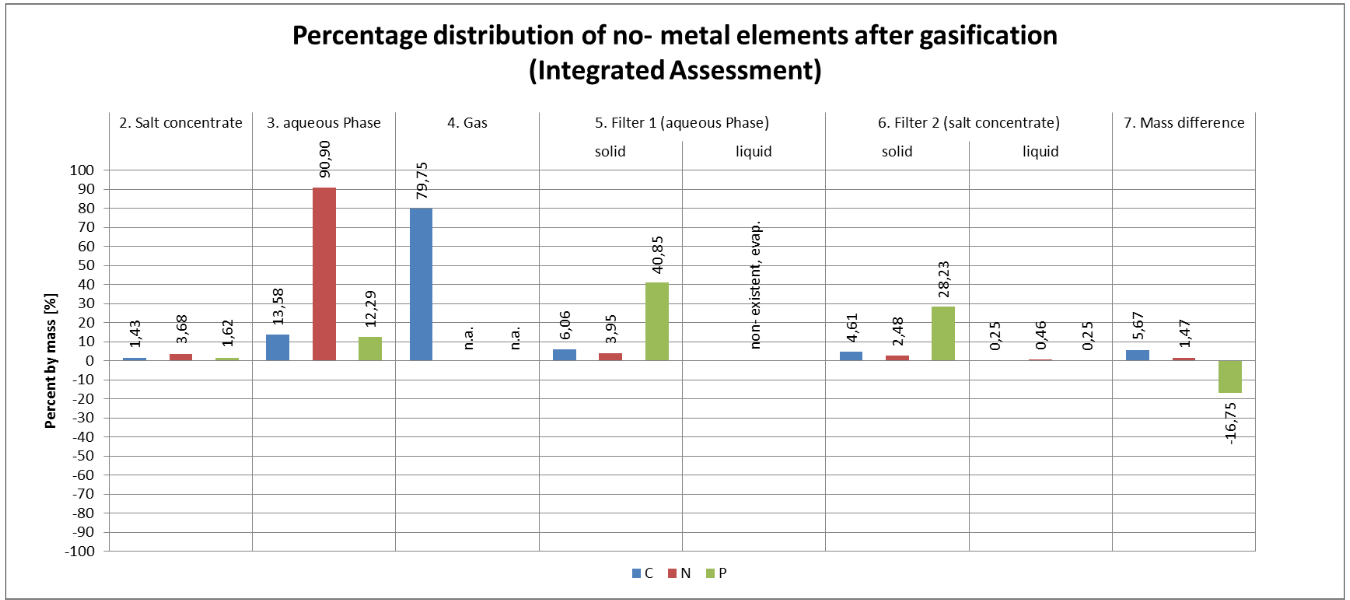
Feed			
HHV:		17302	J/g
Ash content	by 550°C:	22,5	%
	by 815°C:	22,1	%
	by 1000°C:	21,7	%

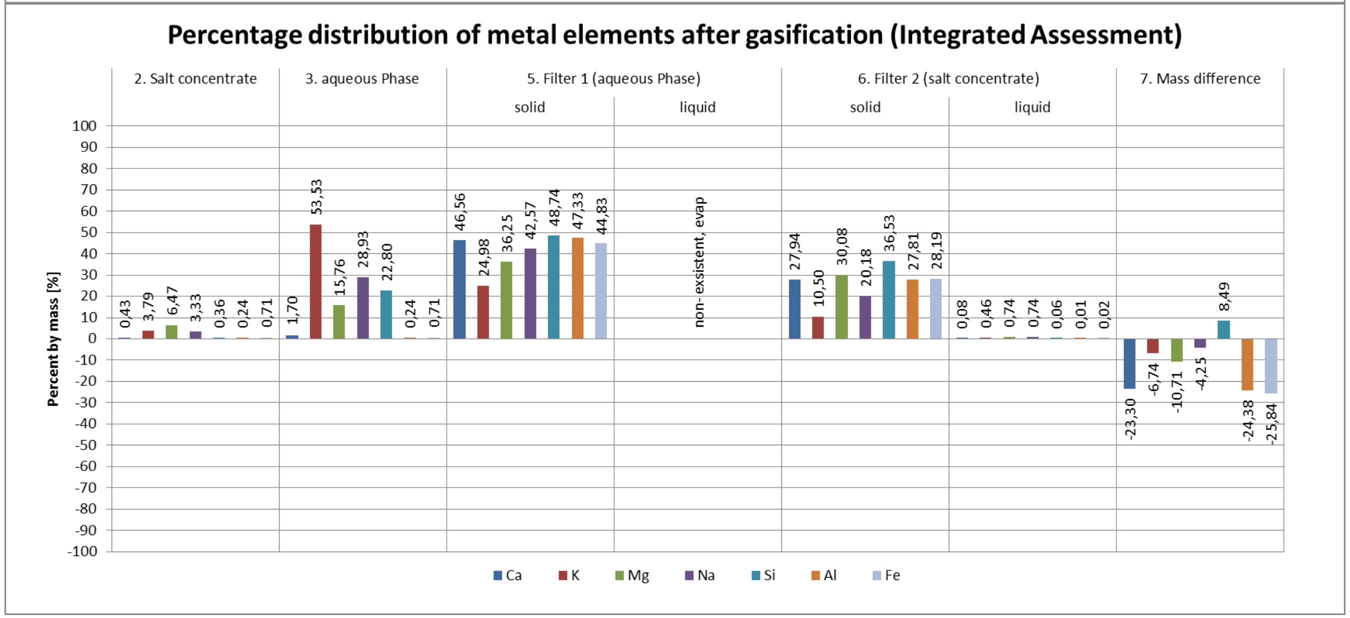
Mass of solids:
Filter1 – aqueous phase m = 48 g
Filter2 – salt concentrate m = 33 g
Method: EDX: O-values; ICP-AAS, except N and except the cursive values in brackets, which were measured by EDX

After the feed phase the gasification of the residues in the reactor proceeds. However the gasification yield cannot be calculated because the amount of dry matter within the reactor is not known and no more carbon is fed. The gas from the residual gasification that leaves the plant contains components from carbon. Even after a long cleaning phase (75 h) with cold water, carbon containing gas (0,94 g) is still discharged.






Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
1,43
3,68
1,62
13,58
90,90
12,29
79,75
n.a.
n.a.
6,06
3,95
40,85
non- existent, evap.
4,61
2,48
28,23
0,25
0,46
0,25
5,67
1,47
-16,75
C
N
P


Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,43
3,79
6,47
3,33
0,36
0,24
0,71
1,70
53,53
15,76
28,93
22,80
0,24
0,71
46,56
24,98
36,25
42,57
48,74
47,33
44,83
non- exsistent, evap
27,94
10,50
30,08
20,18
36,53
27,81
28,19
0,08
0,46
0,74
0,74
0,06
0,01
0,02
-23,30
-6,74
-10,71
-4,25
8,49
-24,38
-25,84
Ca
K
Mg
Na
Si
Al
Fe

2.4.11 Experiment STOWA 11

The influence of the residence time, which was set to 1,7 min, was investigated. This value was the lowest of all tests. Experiment STOWA 11 ran stable.

Experiment conditions		
Dry matter content	12,00	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	655	°C
pressure	280	bar
real residence time	1,70	min
real Feed mass flow	701,0	g/h

Sewage sludge		
time	2,00	h
Total sludge fed	1402	g
C- mass in the total feed*	69,82	g
Total mass effluent	1081	g
Total mass salt brine	59,0	g
TOC-Conc.		
Effluent sample	2,36	mg/g
Salt conc. sample (Position 2)	6,57	mg/g
Gas production	49,15	Nl/h
Conversion of gasification (TC)	71,4	%
TOC-conversion**	94,9	%
Mass-balance	103,6	%
C-balance (TC)	84,3	%
C-deficit (TC)	-11,2	g
Gas composition		
H_2	20,26	Vol %
CO	0,47	Vol %
CO_2	30,30	Vol %
CH_4	34,21	Vol %
C_2H_4	0,94	Vol %
C_2H_6	12,35	Vol %
C_3H_6	0,45	Vol %
C_3H_8	1,03	Vol %
* TIC in the feed not detectable		
** without the C from $KHCO_3$		

STOWA 11									**Filter solid**	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**in the effluent glass jar**	**Filter1 (Position5)**	**Filter2 (Position6)**
Parameter	Concentra-tion dry matter	Concentra-tion diluted sludge*	after 1,54 hours	after 2,54 hours	after 3,54 hours	after experi-ment	after experiment	dark sediment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	weight [%]	[mg/g]	
TC	415	49800	6188	7145	7139	5706	4642	33,8	349	180 *(286)*
TIC	n.d.	n.d.	4062	4593	4729	3728	51	-	n.d.	n.d.
TOC (C)	415	49800	2126	2552	2410	1978	4591	-	349	180 *(286)*
H	60,5	7260	-	-	-	-	-	-	-	-
N	42,2	5064	-	-	-	-	-	-	13,2	10,9
O	-	-	-	-	-	-	-	35,4	359	433
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NH4	-	-	8140	8363	8864	5897	46	-	-	-
TNb	-	-	5479	5652	5641	4730	109	-	-	-
P	24	2880	209	317	378	247	25,1	7,08	54,9	86,5 *(79,4)*
PO4	-	-	476	808	943	639	53,6	-	-	-
S	7,8	936	57,0	77,3	113	328	31,1	1,65	12,2	n.d.
SO4	-	-	148	158	168	133	26,6	-	-	-
Ca	22,5	2700	2,10	3,06	4,20	5,38	8,16	3,29	60,1	78,6
K	3,6	432	1337	1381	1401	1054	81	6,83	30,5	31,9
Mg	3,1	372	4,70	1,60	2,67	3,21	11,1	1,41	5,3	10,8
Na	0,88	106	19,7	23,3	29,9	20,7	4,64	0,49	1,8	1,8
Si	26,5	3180	69,6	58,9	60,4	56,5	19,5	2,69	64	100
Al	12,2	1464	0,15	0,12	0,16	0,7	0,3	1,76	32,3	43
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,32	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	5,6	672	n.d.	n.d.	n.d.	n.d.	0,06	n.d.	1,5	2
Pb	0,11	13,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,48	n.d.
Zn	0,86	103,2	n.d.	n.d.	n.d.	n.d.	0,03	n.d.	2,3	3
Cr	0,039	4,68	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,26	0,18
Fe	13,8	1656	2,88	1,56	1,88	0,85	9,40	4,46	34,4	48,2
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,09	n.d.	0,07	n.d.
Ni	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,12	n.d.	n.d.	n.d.
Cl	1,91	229	45,6	55,0	50,3	136	29,4	0,85	0,27	0,303
Hg	0,0003	0,036	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenol-index	-	-	512	509	513	412	480	-	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Oijen sample 11.06.14

Feed			
HHV:		17302	J/g
Ash content	by 550°C:	22,5	%
	by 815°C:	22,1	%
	by 1000°C:	21,7	%

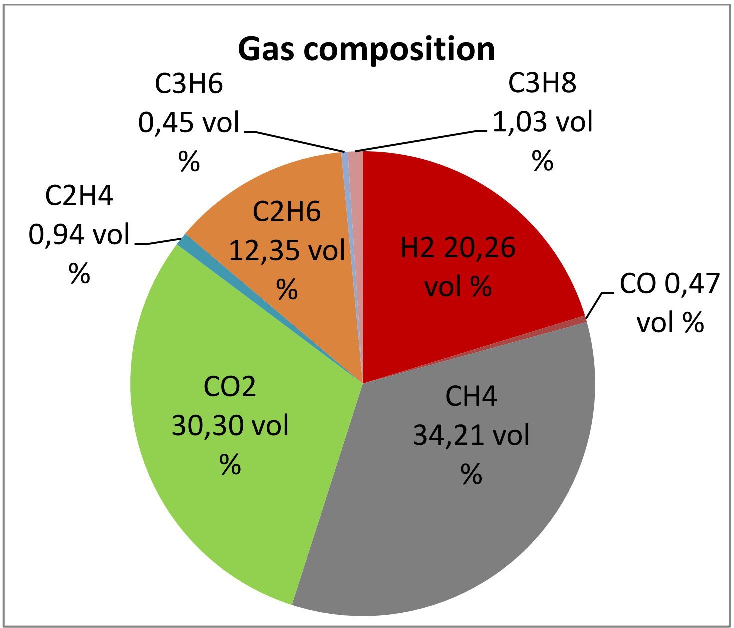
Mass of solids:

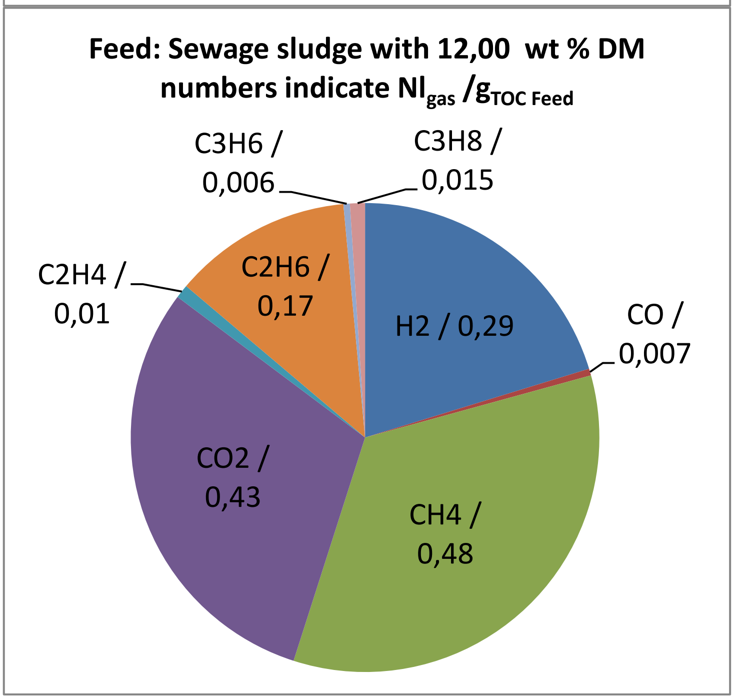
Filter1 – aqueous phase m = 76,3 g

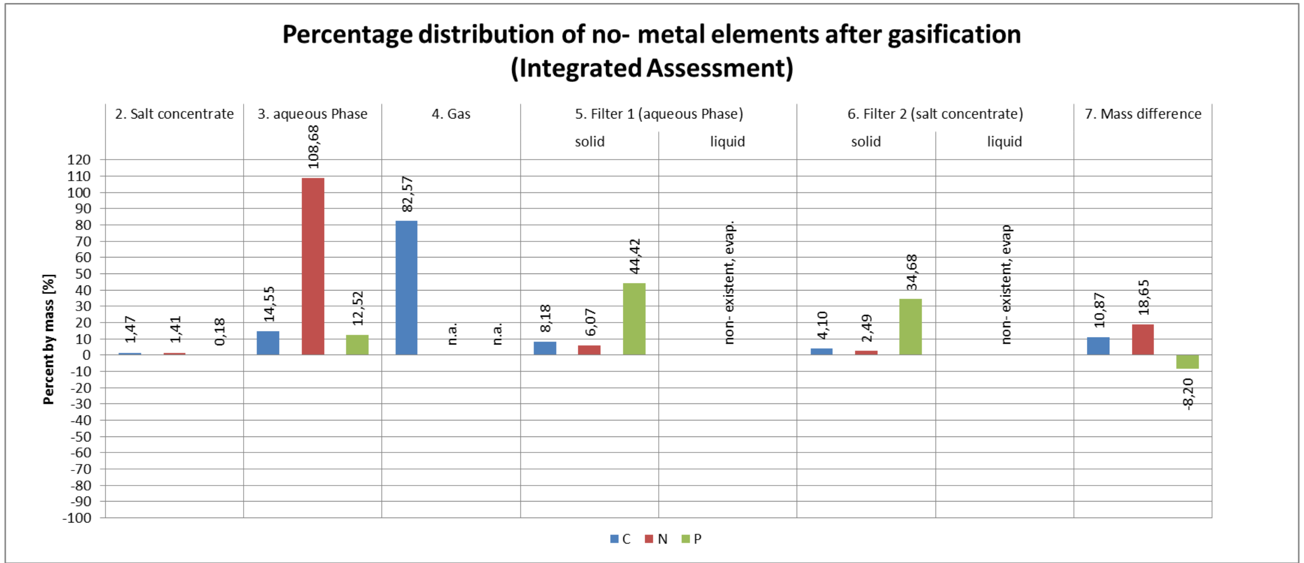
Filter2 – salt concentrate m = 37,8 g

Method: ICP-AAS (except N); EDX: O-values

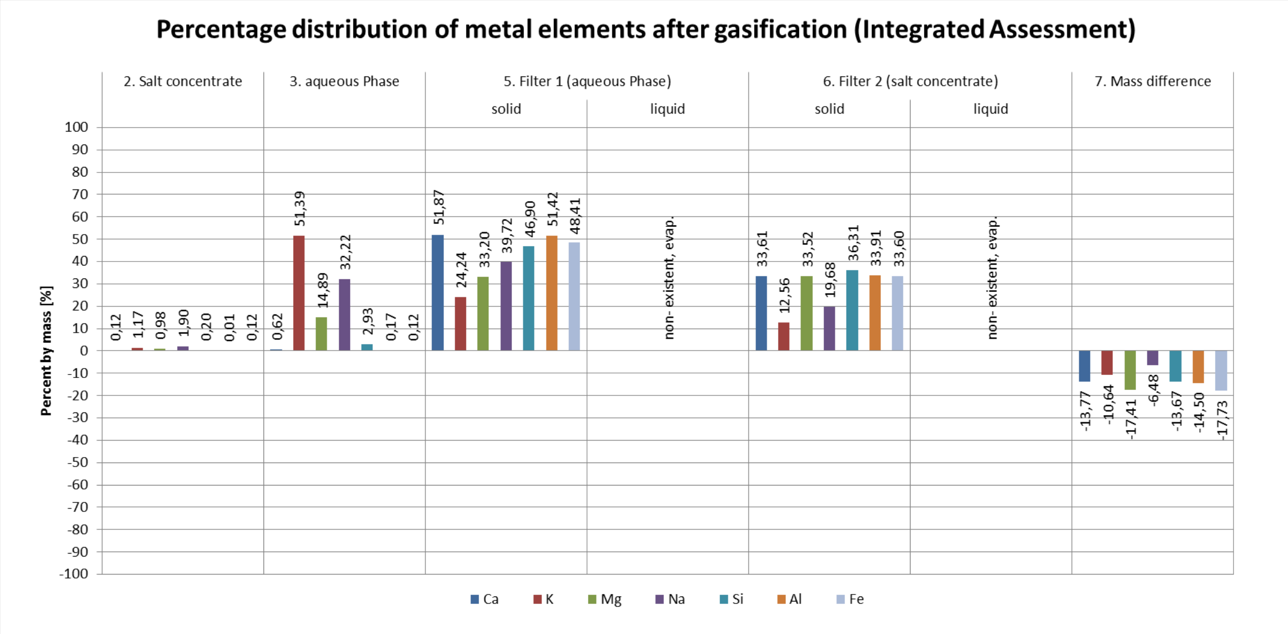
Method: ICP-AAS, except N and except the cursive values in brackets, which were measured by EDX







Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
solid
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
1,47
1,41
0,18
14,55
108,68
12,52
82,57
n.a.
n.a.
8,18
6,07
44,42
non- existent, evap.
4,10
2,49
34,68
non- existent, evap
10,87
18,65
-8,20
C
N
P



Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
solid
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
0,12
1,17
0,98
1,90
0,20
0,01
0,12
0,62
51,39
14,89
32,22
2,93
0,17
0,12
51,87
24,24
33,20
39,72
46,90
51,42
48,41
non- existent, evap.
33,61
12,56
33,52
19,68
36,31
33,91
33,60
non- existent, evap.
-13,77
-10,64
-17,41
-6,48
-13,67
-14,50
-17,73
Ca
K
Mg
Na
Si
Al
Fe

2.4.12 Experiment STOWA 12

The temperature of the preheater was lowered (450 °C) to investigate the influence on the separation of the salts and a high residence time (5,31 min) was reached. Experiment STOWA 12 ran stable.

Experiment conditions		
Dry matter content	12,42	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	652	°C
pressure	280	bar
real residence time	5,31	min
real Feed mass flow	215,2	g/h

Sewage sludge		
time	2,50	h
Total sludge fed	538	g
C- mass in the total feed*	27,73	g
Total mass effluent	270	g
Total mass salt brine	63,7	g
TOC-Conc.		
Effluent sample	1,15	mg/g
Salt conc. sample (Position 2)	0,79	mg/g
Gas production	16,74	Nl/h
Conversion of gasification (TC)	75,7	%
TOC-conversion**	97,9	%
Mass-balance	108,1	%
C-balance (TC)	86,0	%
C-deficit (TC)	-3,9	g
Gas composition		
H_2	18,96	Vol %
CO	0,32	Vol %
CO_2	32,52	Vol %
CH_4	35,29	Vol %
C_2H_4	0,23	Vol %
C_2H_6	12,57	Vol %
C_3H_6	0,02	Vol %
C_3H_8	0,09	Vol %
* TIC in the feed not detectable		
** without the C from $KHCO_3$		

STOWA 12									Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis** (Position 2)	**in the effluent glass jar** (after cold pipe-cleaning)	**Filter1** (Position 5)	**Filter2** (Position 6)
Parameter	Concentra-tion dry matter	Concentra-tion diluted sludge*	after 7,33 hours	after 8,33 hours	after 9,33 hours	after experiment	after experiment	dark sediment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	weight [%]	[mg/g]	
TC	415	51543	5227	6067	7254	4567	1406	17,2	155	137 *(166)*
TIC	n.d.	n.d.	4177	4818	6088	3790	n.d.	-	n.d.	n.d.
TOC (C)	415	51543	1049	1249	1166	776	1404	-	155	137 *(166)*
H	60,5	7514	-	-	-	-	-	-	-	-
N	42,2	5241	-	-	-	-	-	-	n.d.	n.d.
O	-	-	-	-	-	-	-	41,5	421	430
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NH4	-	-	9665	10099	9626	5982	10	-	-	-
TNb	-	-	7016	6619	6901	4963	274	-	-	-
P	24	2981	104	110	113	70,6	20,2	9,34	87,8	88,4 *(101,8)*
PO4	-	-	264	283	284	40,6	32,9	-	-	-
S	7,8	969	55,3	55,0	55,5	31,3	48,1	0,84	n.d.	n.d.
SO4	-	-	66,3	63,6	62,6	46,0	37,9	-	-	-
Ca	22,5	2795	4,62	4,85	4,99	5,61	15,9	8,09	89,9	80,5
K	3,6	447	767	772	725	437	137	5,49	51,3	49,5
Mg	3,1	385	4,17	5,46	13,4	11,7	13	0,91	10,7	10,6
Na	0,88	109	9,54	9,80	9,77	6,22	8,44	0,22	3,3	2,8
Si	26,5	3291	66,5	64,1	66,5	47,9	14,3	6,24	66,5	85,6
Al	12,2	1515	0,32	0,31	0,31	0,31	0,78	4,44	50,6	41,8
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	5,6	696	n.d.	n.d.	n.d.	n.d.	n.d.	0,33	2,2	2,1
Pb	0,11	13,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,86	107	n.d.	n.d.	n.d.	n.d.	0,08	0,32	3,4	3,2
Cr	0,039	4,84	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,32	0,27
Fe	13,8	1714	2,58	2,97	3,51	1,97	10,70	4,54	48,8	49,3
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,44	n.d.
Ni	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1	n.d.
Cl	1,91	237	41,46	40,18	39,7	41	32,90	0,21	1,3	1,3
Hg	0,0003	0,0373	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	236	238	232	134	13	-	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Oijen sample 11.06.14

Feed			
HHV:		17302	J/g
Ash content	by 550°C:	22,5	%
	by 815°C:	22,1	%
	by 1000°C:	21,7	%

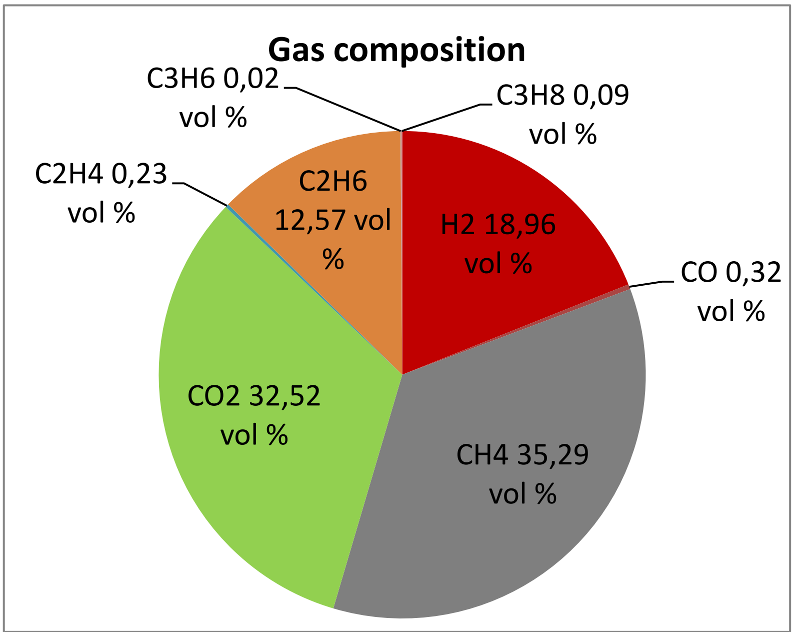
<u>Mass of solids:</u>

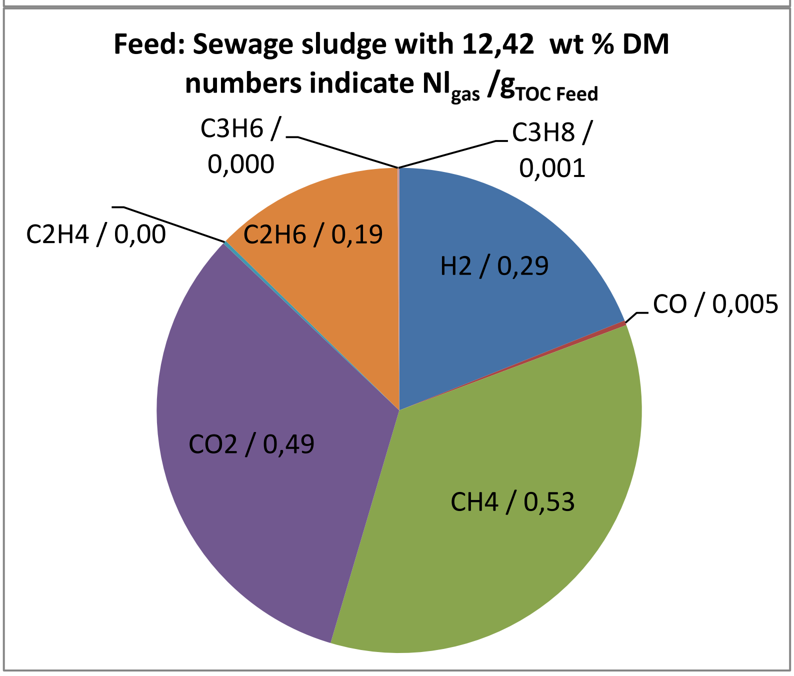
Filter1 – aqueous phase m = 4,9 g

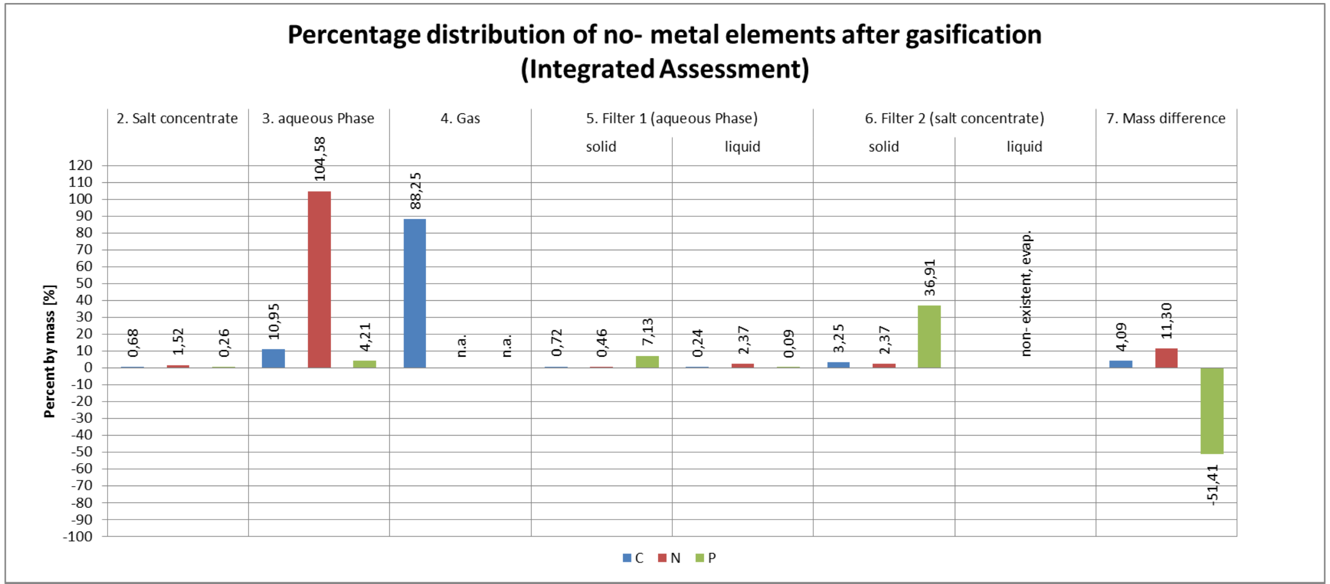
Filter2 – salt concentrate m = 25,2 g

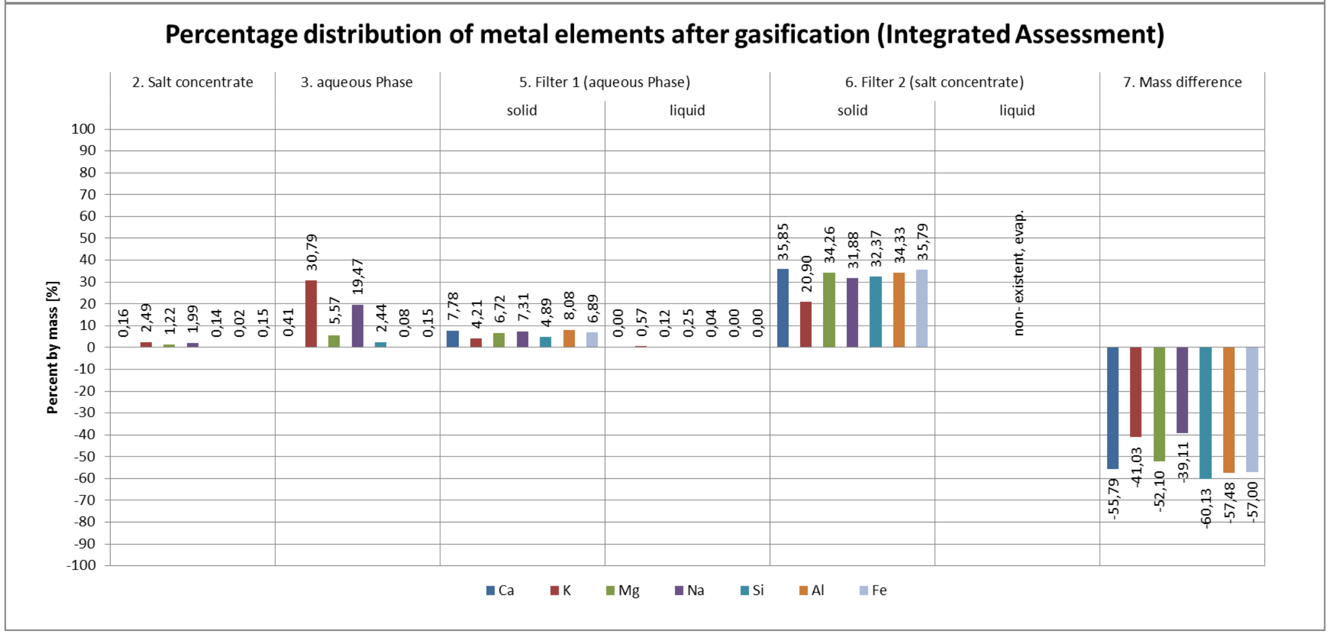
Method: EDX: Cl and O-values;

ICP-AAS, except N and except the cursive values in brackets, which were measured by EDX






Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,68
1,52
0,26
10,95
104,58
4,21
88,25
n.a.
n.a.
0,72
0,46
7,13
0,24
2,37
0,09
3,25
2,37
36,91
non- existent, evap.
4,09
11,30
-51,41
C
N
P


Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,16
2,49
1,22
1,99
0,14
0,02
0,15
0,41
30,79
5,57
19,47
2,44
0,08
0,15
7,78
4,21
6,72
7,31
4,89
8,08
6,89
0,00
0,57
0,12
0,25
0,04
0,00
0,00
35,85
20,90
34,26
31,88
32,37
34,33
35,79
non- existent, evap.
-55,79
-41,03
-52,10
-39,11
-60,13
-57,48
-57,00
Ca
K
Mg
Na
Si
Al
Fe

The recovery rate of the trace elements is very poor. This is due to the very low total amount present in the feed treated. To give a more detailed picture the balance of Cu, Pb, Zn and Hg for the experiments 13 to 17 is given. Other elements like Fe, Al, Ca and P are illustrated in the diagrams and/or listed in the Appendix.

Cu-balance		
	[g Cu]	[%]
Cu- amount feed:	1,41	100
Cu- amount outlet + cold water cleaning phase + V41:	0	0
Cu- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0,0108	0,765
filter F01 - liquid:	0,0000088	0,000624
filter F02 - salt separation system / solid:	0,0529	3,75
filter F02 - salt separation system / liquid:	0	0
Cu- amount result:	0,0637	4,52
deficit:	-1,34	-95,5
Pb-balance		
	[g Pb]	[%]
Pb- amount feed:	0,0277	100
Pb- amount outlet + cold water cleaning phase + V41:	0	0
Pb- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0	0
filter F01 - liquid:	0,000044	0,159
filter F02 - salt separation system / solid:	0	0
filter F02 - salt separation system / liquid:	0	0
Pb- amount result:	0,000044	0,159
deficit:	-0,0276	-99,8
Zn-balance		
	[g Zn]	[%]
Zn- amount feed:	0,216	100
Zn- amount outlet + cold water cleaning phase + V41:	0	0
Zn- amount salt concentrate + cold water cleaning phase + V33:	0,0000187	0,00865
filter F01 - solid:	0,0167	7,70
filter F01 - liquid:	0,0000088	0,00407
filter F02 - salt separation system / solid:	0,0806	37,3
filter F02 - salt separation system / liquid:	0	0
Zn- amount result:	0,0973	45,0
deficit:	-0,119	-55,0
Hg-balance		
	[g Hg]	[%]
Hg- amount feed:	0,0000755	100
Hg- amount outlet + cold water cleaning phase + V41:	0	0
Hg- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0	0
filter F01 - liquid:	0,000044	58,3
filter F02 - salt separation system / solid:	0	0
filter F02 - salt separation system / liquid:	0	0
Hg- amount result:	0,000044	58,3
deficit:	-0,0000315	-41,7
As and Cd are not detectable in the feed		

2.4.13 Experiment STOWA 13

In this experiment sewage sludge with a low dry matter content of 8,59 wt.-% was used to investigate especially its influence on the conversion of gasification. Experiment 13 ran stable.

Experiment conditions		
Dry matter content	8,59	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	653	°C
pressure	280	bar
real residence time	2,92	min
real Feed mass flow	420,8	g/h

Sewage sludge		
time	4,00	h
Total sludge fed	1683	g
C- mass in the total feed*	61,01	g
Total mass effluent	1269	g
Total mass salt brine	27,5	g
TOC-Conc.		
Effluent sample	0,82	mg/g
Salt conc. sample (Position 2)	0,30	mg/g
Gas production	26,37	Nl/h
Conversion of gasification (TC)	77,3	%
TOC-conversion**	97,9	%
Mass-balance	101,4	%
C-balance (TC)	87,1	%
C-deficit (TC)	-8,1	g
Gas composition		
H_2	27,46	Vol %
CO	0,17	Vol %
CO_2	32,86	Vol %
CH_4	28,26	Vol %
C_2H_4	0,32	Vol %
C_2H_6	10,45	Vol %
C_3H_6	0,07	Vol %
C_3H_8	0,42	Vol %

* TIC in the feed not detectable
** without the C from $KHCO_3$

STOWA 13									Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**in the effluent glass jar** (after experiment)	**Filter1** (Position 5)	**Filter2** (Position 6)
Parameter	Concentra-tion dry matter	Concen-tration diluted sludge*	after 6 hours	after 7 hours	after 8 hours	after experi-ment	after experiment	dark sediment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	weight [%]	[mg/g]	
TC	422	36250	3844	3925	3946	2687	388	8,17	110	201 *(136)*
TIC	n.d.	n.d.	3023	3080	3090	2171	65	-	n.d.	n.d.
TOC (C)	422	36250	821	845	856	517	323	-	110	201 *(136)*
H	60,9	5231	-	-	-	-	-	-	-	-
N	48,2	4140	-	-	-	-	-	-	n.d.	n.d.
O	-	-	-	-	-	-	-	39,2	413	400
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-	-
NH4	-	-	6309	6856	6771	4321	67	-	-	-
TNb	-	-	4103	4009	3304	2098	100	-	-	-
P	20	1718	186	191	188	134	12,6	9,74	79,2	77*(70,9)*
PO4	-	-	479	479	481	51,4	33,3	-	-	-
S	n.d.	n.d.	32,2	44,8	36,1	23,0	29,4	1,86	3,7	3,8
SO4	-	-	100	104	102	79,0	48,1	-	-	-
Ca	18,5	1589	3,09	3,30	3,06	4,59	5,0	7,75	73,8	53,6
K	3,2	275	1263	1263	1260	874	149	7,97	51,5	29,6
Mg	3,1	266	1,05	1,59	1,49	5,61	8,83	1,04	10,4	10,8
Na	1	85,9	5,70	6,10	5,95	4,83	3,4	0,36	4,1	2
Si	40,6	3488	65,4	61,1	62,3	72,5	9,5	6,15	88,9	67,2
Al	12,5	1074	0,33	0,33	0,31	0,34	0,39	3,72	48,8	41,6
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,04	3,44	n.d.	n.d.	n.d.	n.d.	n.d.	0,32	4,4	3,4
Pb	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,9	77,3	0,03	0,03	0,03	n.d.	n.d.	0,28	3,1	3,3
Cr	0,15	12,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Fe	13,6	1168	0,88	1,13	0,72	0,88	1,98	12,65	106	104
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,29	n.d.	n.d.	n.d.
Ni	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cl	2,02	174	49,7	52,5	49,0	51	33,3	0,48	n.d.	n.d.
Hg	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	128	135	140	97	20	-	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Oijen sample 03.07.14

Feed			
HHV:		17426	J/g
Ash content	by 550°C:	20,8	%
	by 815°C:	20,4	%
	by 1000°C:	20,01	%

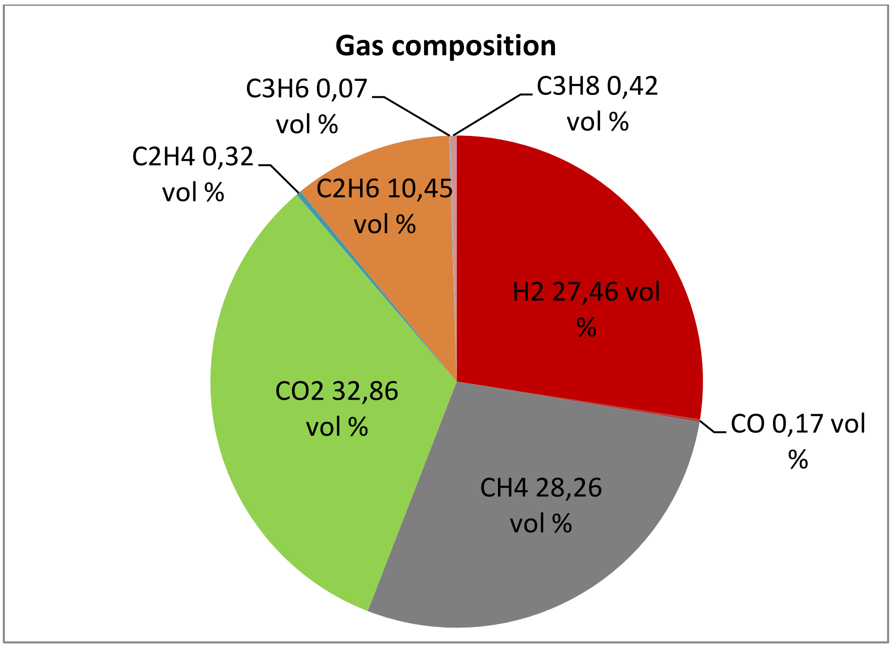
Mass of solids:

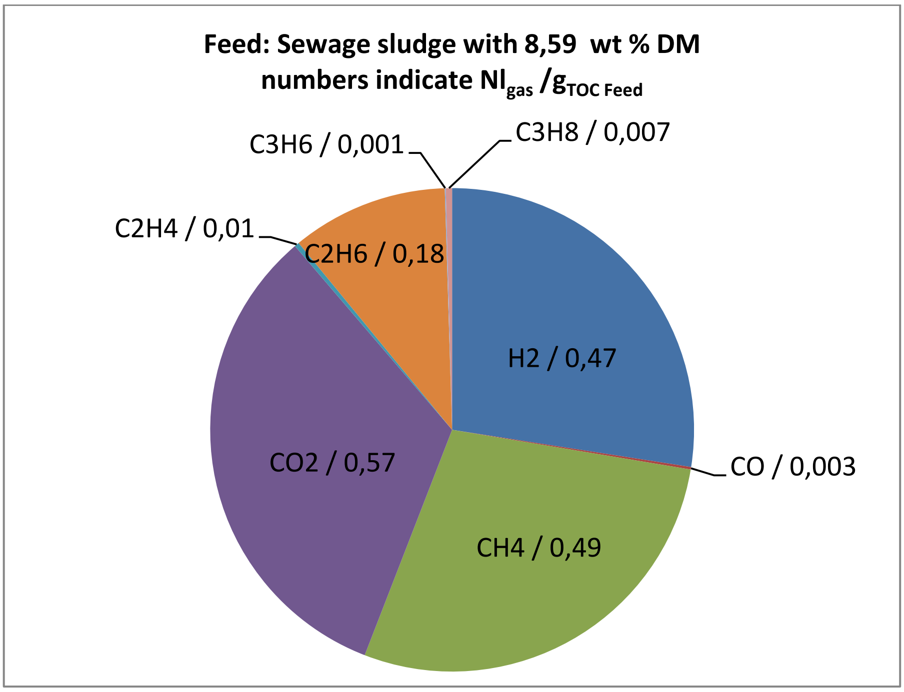
Filter1 – aqueous phase m = 21,2 g

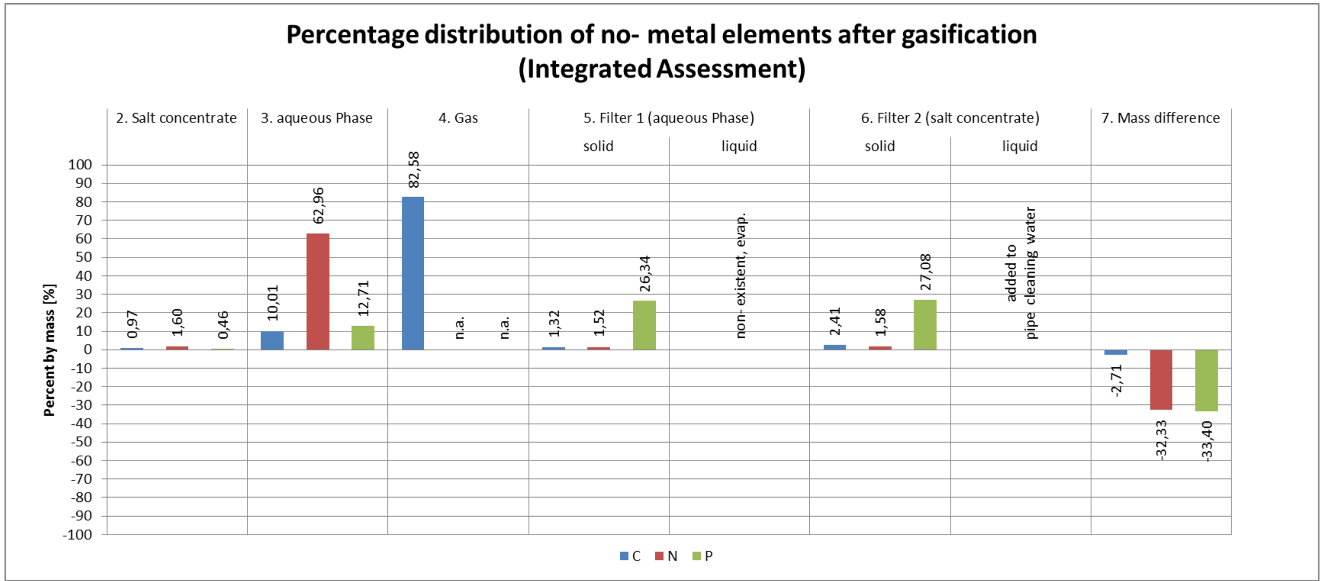
Filter2 – salt concentrate m = 22,1 g

Method: EDX (TIC was determined by another method and <0,1 wt. %)

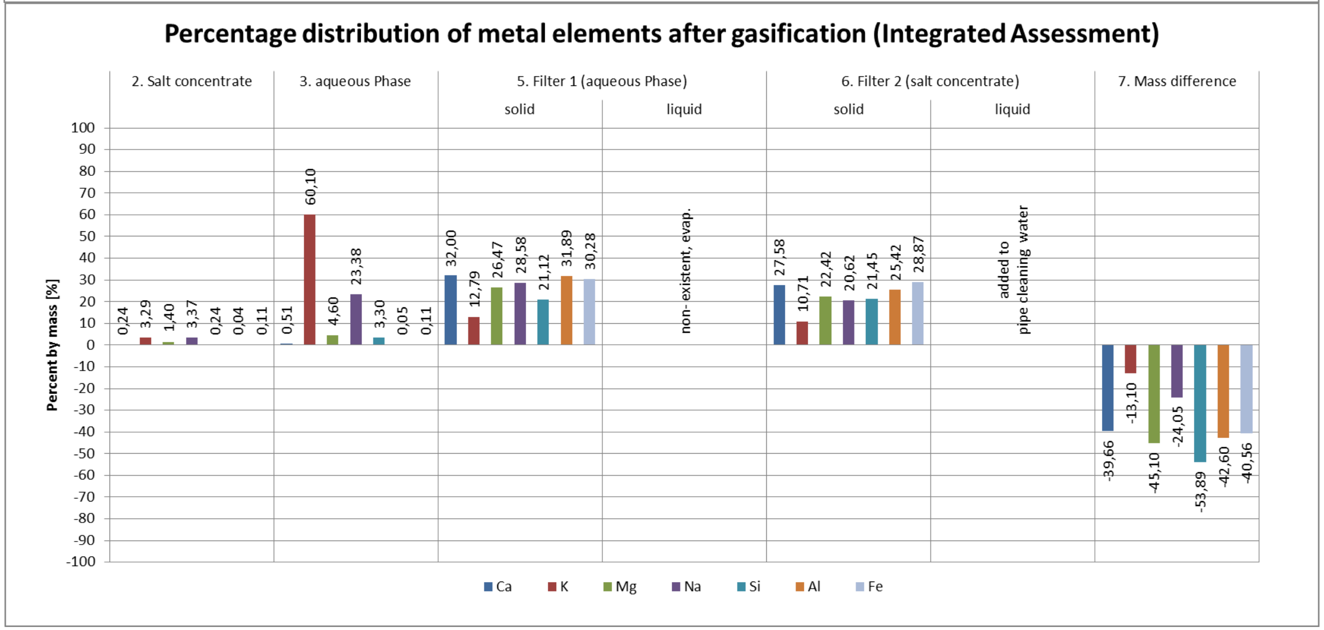
(P) the cursive values in brackets by ICP-AAS; (C) the cursive values in brackets by another method.







Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,97
1,60
0,46
10,01
62,96
12,71
82,58
n.a.
n.a.
1,32
1,52
26,34
non- existent, evap.
2,41
1,58
27,08
added to pipe cleaning water
-2,71
-32,33
-33,40
C
N
P



Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,24
3,29
1,40
3,37
0,24
0,04
0,11
0,51
60,10
4,60
23,38
3,30
0,05
0,11
32,00
12,79
26,47
28,58
21,12
31,89
30,28
non- existent, evap.
27,58
10,71
22,42
20,62
21,45
25,42
28,87
added to pipe cleaning water
-39,66
-13,10
-45,10
-24,05
-53,89
-42,60
-40,56
Ca
K
Mg
Na
Si
Al
Fe

As, Cd, Hg and Pb could not be detected in the feed and in the product streams.

Cu-balance		
	[g Cu]	[%]
Cu- amount feed:	0,0116	100
Cu- amount outlet + cold water cleaning phase + V41:	0	0
Cu- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0,0488	421
filter F01 - liquid:	0	0
filter F02 - salt separation system / solid:	0,0464	401
filter F02 - salt separation system / liquid:	0	0
Cu- amount result:	0,0952	822
deficit:	0,0836	722
Zn-balance		
	[g Zn]	[%]
Zn- amount feed:	0,26	100
Zn- amount outlet + cold water cleaning phase + V41:	0,000038	0,0146
Zn- amount salt concentrate + cold water cleaning phase + V33:	0,000144	0,0552
filter F01 - solid:	0,0827	31,6
filter F01 - liquid:	0	0
filter F02 - salt separation system / solid:	0,0751	28,9
filter F02 - salt separation system / liquid:	0	0
Zn- amount result:	0,158	60,7
deficit:	-0,102	-39,3

2.4.14 Experiment STOWA 14

In experiment STOWA 14 no KHCO3 was added. The result was clogging after 2 h. Then the experiment was restarted and regularly stopped after 10 h in total. Generally this experiment shows frequently delta p in the reaction system and it was necessary to separate more salt concentrate than planned.

Experiment conditions		
Dry matter content	12,81	%
K^+-conc. ($KHCO_3$)	without	mg/l
real Temperature	654	°C
pressure	280	bar
real residence time	2,70	min
real Feed mass flow	430,5	g/h

Sewage sludge		
time	2,00	h
Total sludge fed	861	g
C- mass in the total feed*	46,54	g
Total mass effluent	501	g
Total mass salt brine	52,9	g
TOC-Conc.		
Effluent sample	2,82	mg/g
Salt conc. sample (Position 2)	4,49	mg/g
Gas production	27,75	Nl/h
Conversion of gasification (TC)	63,35	%
TOC-conversion	94,70	%
Mass-balance	104,37	%
C-balance (TC)	75,10	%
C-deficit (TC)	-11,59	g
Gas composition		
H_2	12,63	Vol %
CO	5,62	Vol %
CO_2	39,19	Vol %
CH_4	32,04	Vol %
C_2H_4	0,60	Vol %
C_2H_6	9,61	Vol %
C_3H_6	0,12	Vol %
C_3H_8	0,19	Vol %
* TIC in the feed not detectable		

No KHCO3 was added to the feed and therefor the concentration of potassium in this process is generally lower.

STOWA 14									Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**		**Filter1 (Position 6)**	**Filter2 (Position 5)**
Parameter	Concentration dry matter	Concentration diluted sludge*	after 3 hours	after 4 hours	after 5 hours	after experiment	after experiment		Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[mg/g]	
TC	422	54058	6672	6628	7001	5498	4707		332	208 *(225)*
TIC	n.d.	n.d.	3993	3856	4002	3120	214		n.d.	n.d.
TOC (C)	422	54058	2679	2772	2999	2378	4493		332	208 *(225)*
H	60,9	7801	-	-	-	-	-		-	-
N	48,2	6174	-	-	-	-	-		16,4	11,5
O	-	-	-	-	-	-	-		344	423
NO3	-	-	n.d.	n.d.	n.d.	29,2	n.d.		-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.		-	-
NH4	-	-	5966	6399	6222	5484	993		-	-
TNb	-	-	5910	5567	5982	4642	1584		-	-
P	20	2562	50,9	57,3	57,0	47,0	42,1		62,4	69,4 *(89,3)*
PO4	-	-	121	131	141	115	105		-	-
S	n.d.	n.d.	104	151	150	109	167		14,6	6,6
SO4	-	-	60,0	58,0	62,4	48,6	145		-	-
Ca	18,5	2370	9,50	11,5	10,8	10,3	30,6		59,9	67,3
K	3,2	410	130	143	142	113	159		6,7	6,14
Mg	3,1	397	4,95	5,45	4,90	8,92	13,5		8,3	9,9
Na	1	128	23,1	25,9	26,2	20,1	26,2		1,39	1,08
Si	40,6	5201	45,9	46,0	47,1	46,6	90,7		96,7	110
Al	12,5	1601	0,08	n.d.	0,08	0,05	0,54		38,5	44,1
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		0,02	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.
Cu	0,04	5,12	n.d.	n.d.	n.d.	n.d.	n.d.		1,71	1,89
Pb	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		0,349	0,385
Zn	0,9	115	n.d.	n.d.	n.d.	n.d.	n.d.		2,58	3,01
Cr	0,15	19,2	n.d.	n.d.	n.d.	n.d.	n.d.		1,97	0,176
Fe	13,6	1742	0,07	n.d.	0,07	0,06	2,24		45,5	49,9
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		0,035	0,027
Ni	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		34,70	0,19
Cl	2,02	259	77,1	77,3	79,5	94,1	87,5		0,40	0,44
Hg	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		0,00241	0,00087
Phenolindex	-	-	423	438	450	370	87		-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Oijen sample 03.07.14

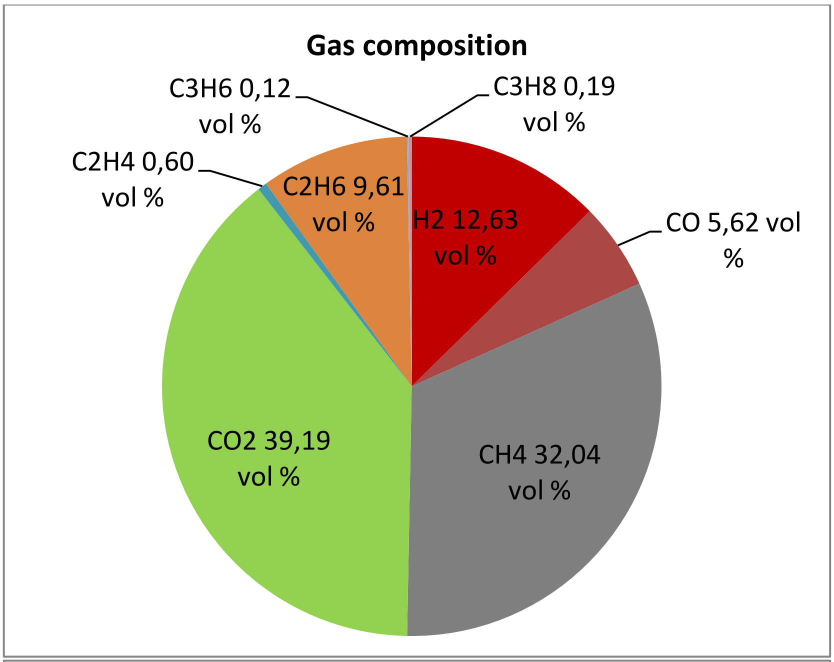
Feed			
HHV:		17426	J/g
Ash content	by 550°C:	20,8	%
	by 815°C:	20,4	%
	by 1000°C:	20,01	%

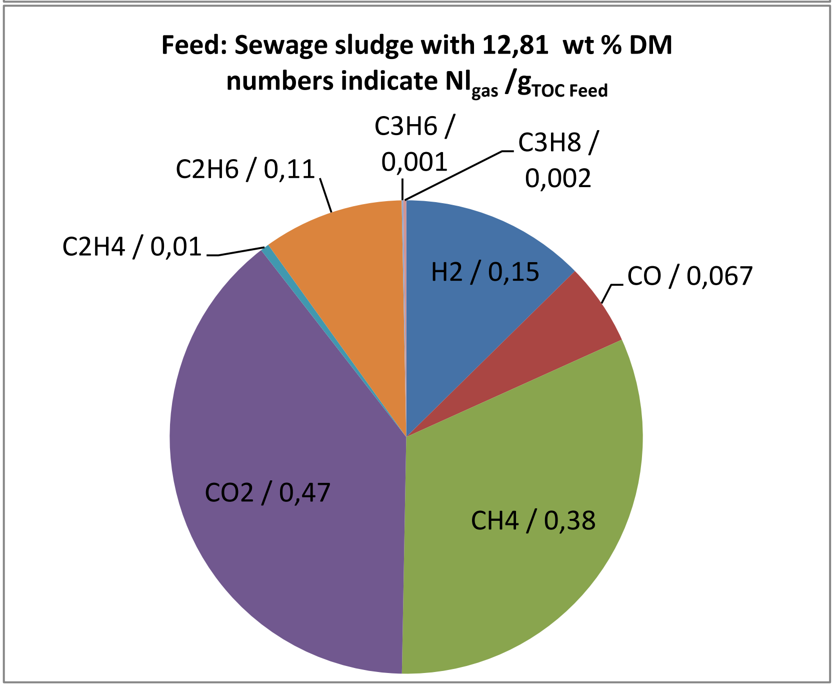
<u>Mass of solids:</u>

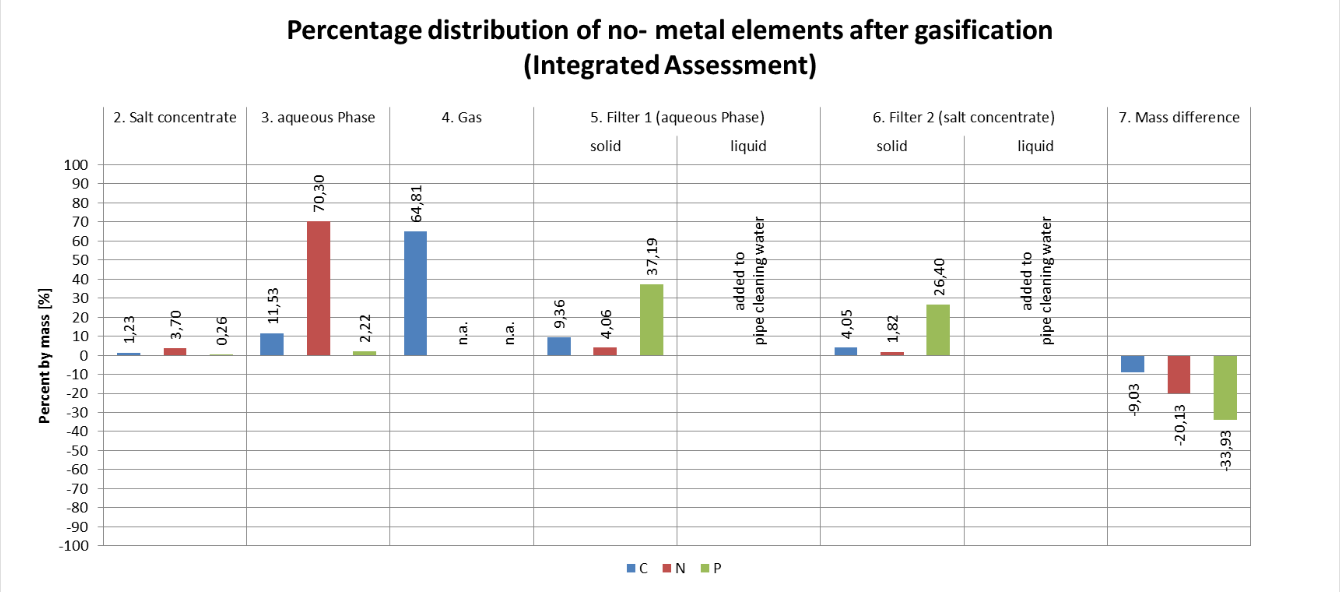
Filter1 – aqueous phase m = 47 g

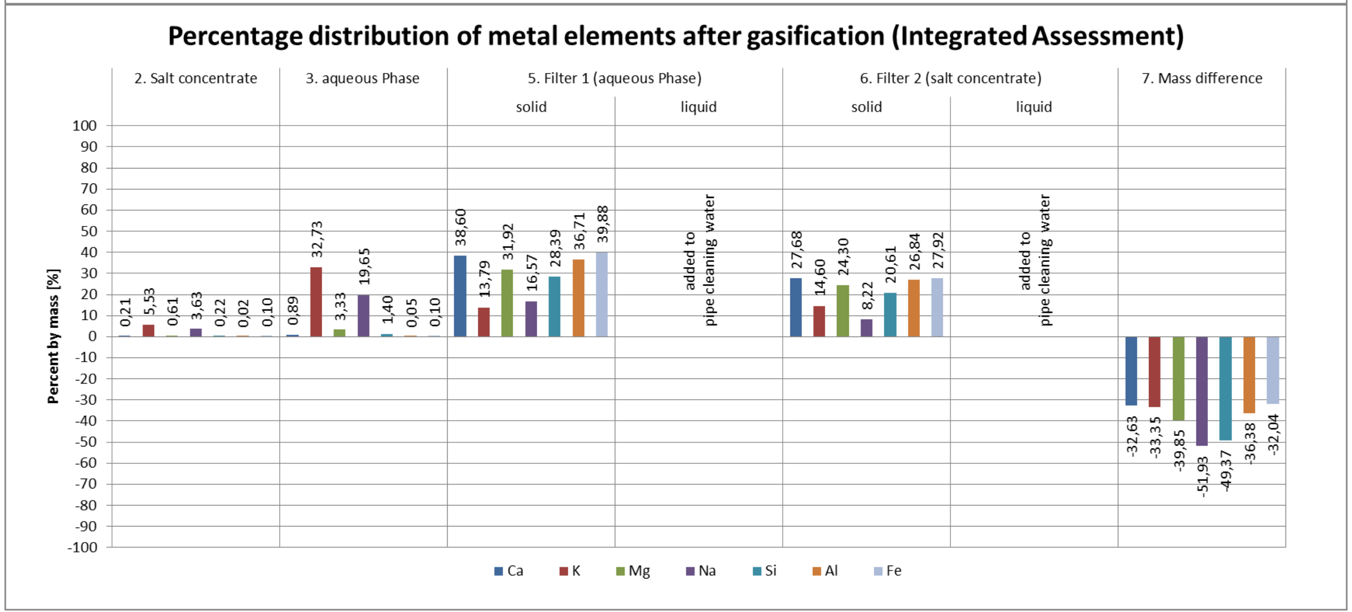
Filter2 – salt concentrate m = 30 g

Method: ICP-AAS, except N and except the cursive values in brackets, which were measured by EDX EDX: O-values






Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
1,23
3,70
0,26
11,53
70,30
2,22
64,81
n.a.
n.a.
9,36
4,06
37,19
added to pipe cleaning water
4,05
1,82
26,40
added to pipe cleaning water
-9,03
-20,13
-33,93
C
N
P


Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,21
5,53
0,61
3,63
0,22
0,02
0,10
0,89
32,73
3,33
19,65
1,40
0,05
0,10
38,60
13,79
31,92
16,57
28,39
36,71
39,88
added to pipe cleaning water
27,68
14,60
24,30
8,22
20,61
26,84
27,92
added to pipe cleaning water
-32,63
-33,35
-39,85
-51,93
-49,37
-36,38
-32,04
Ca
K
Mg
Na
Si
Al
Fe

2.4.15 Experiment STOWA 15

The intention of the test was the investigation of the salt separation at low temperature. Therefore the preheater temperature was set to only 400 °C the lowest value of all experiments. Experiment STOWA 15 ran stable.

Experiment conditions		
Dry matter content	13,35	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	653	°C
pressure	280	bar
real residence time	2,66	min
real Feed mass flow	430	g/h

Sewage sludge		
time	3,00	h
Total sludge fed	1290	g
C- mass in the total feed*	69,23	g
Total mass effluent	883	g
Total mass salt brine	154	g
TOC-Conc.		
Effluent sample	2,53	mg/g
Salt conc. sample (Position 2)	1,83	mg/g
Gas production	29,23	Nl/h
Conversion of gasification (TC)	65,3	%
TOC-conversion**	95,6	%
Mass-balance	105,9	%
C-balance (TC)	78,7	%
C-deficit (TC)	-14,9	g
Gas composition		
H_2	16,05	Vol %
CO	0,50	Vol %
CO_2	37,32	Vol %
CH_4	33,86	Vol %
C_2H_4	0,53	Vol %
C_2H_6	11,24	Vol %
C_3H_6	0,14	Vol %
C_3H_8	0,35	Vol %
* TIC in the feed not detectable		
** without the C from $KHCO_3$		

STOWA 15									Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**		**Filter1 (Position 5)**	**Filter2 (Position 6)**
Parameter	Concentration dry matter	Concentration diluted sludge*	after 3 hours	after 4 hours	after 5 hours	after experiment	after experiment		Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[mg/g]	
TC	402	53667	8667	8462	8040	6330	2506		343	248 *(279)*
TIC	n.d.	n.d.	5940	5803	5565	4429	269		n.d.	n.d.
TOC (C)	402	53667	2727	2659	2475	1901	2236		343	248 *(279)*
H	60,6	8090	-	-	-	-	-		-	-
N	47,4	6328	-	-	-	-	-		n.d.	16,2
O	-	-	-	-	-	-	-		326	391
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.		-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.		-	-
NH4	-	-	6702	6920	6022	1764	382		-	-
TNb	-	-	8100	7599	7613	6025	567		-	-
P	21,8	2910	178	280	242	141	23,6		60,9	76,9 *(82,9)*
PO4	-	-	477	747	631	68,7	44,4		-	-
S	8,7	1161	493	581	703	446	574		38,4	21,1
SO4	-	-	150	167	127	91,0	67,4		-	-
Ca	16,3	2176	7,15	11,9	14,2	9,55	13		53,4	57
K	2,96	395	966	1129	842	588	182		44,8	30,3
Mg	2,86	381	2,20	1,30	1,30	4,5	9		8,8	11,9
Na	0,84	112	6,60	12,3	17,7	8	6,45		3,4	2,8
Si	35,3	4713	56,3	47,5	56,9	59,3	32,3		128	141
Al	12,2	1629	0,08	0,07	0,07	0,05	0,18		42,2	43,3
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.
Cu	0,59	78,8	n.d.	n.d.	n.d.	n.d.	n.d.		2,2	2,3
Pb	0,111	14,8	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.
Zn	0,96	128	n.d.	n.d.	n.d.	n.d.	n.d.		3,6	3,7
Cr	0,042	5,61	n.d.	n.d.	n.d.	n.d.	n.d.		0,17	0,22
Fe	20,9	2790	0,02	0,06	1,94	0,27	16,4		76,7	84,3
Mo	0,015	2,00	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.
Ni	0,024	3,20	n.d.	n.d.	n.d.	n.d.	n.d.		1,3	n.d.
Cl	2,83	378	70,6	83,5	75,8	69	44,4		0,0818	0,401
Hg	0,00063	0,084	0,0	0,0	0,0	0,0	0,0		n.d.	n.d.
Phenolindex	-	-	506	535	518	365	36		-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Oijen sample 18.07.14

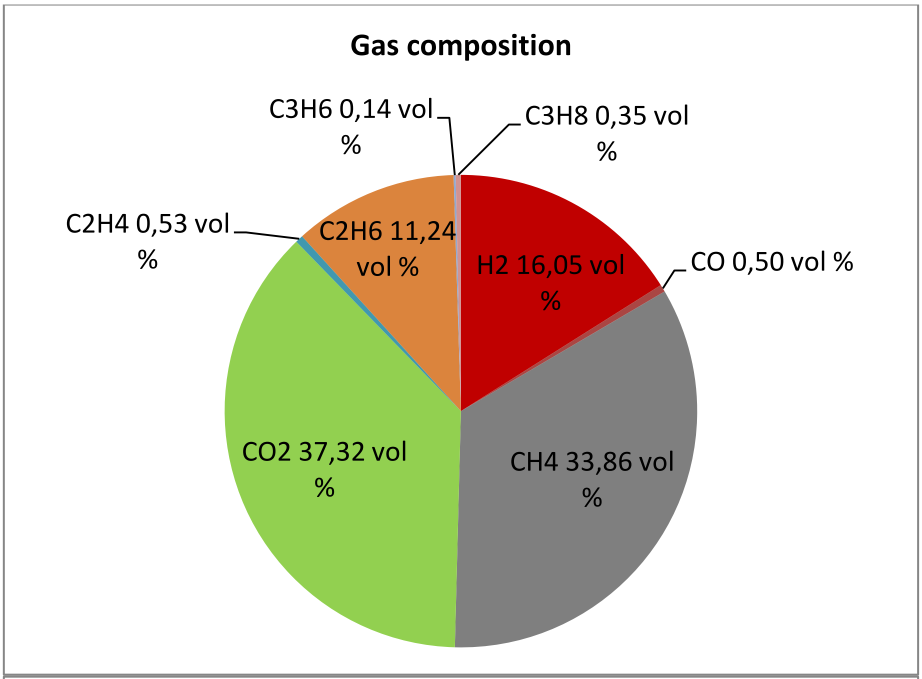
Feed			
HHV:		16504	J/g
Ash content	by 550°C:	22,45	%
	by 815°C:	22,17	%
	by 1000°C:	21,82	%

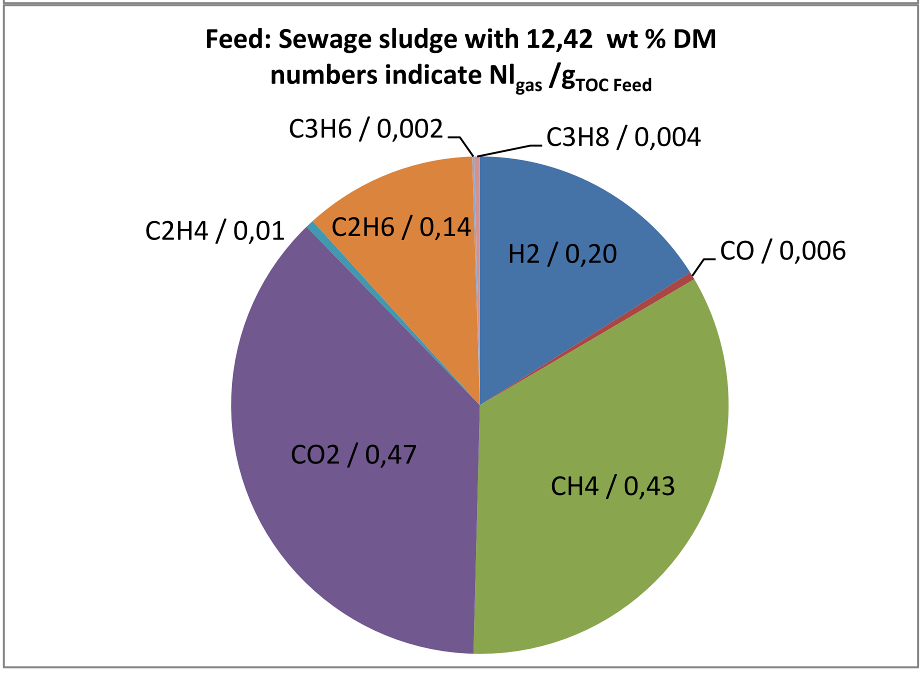
Mass of solids:

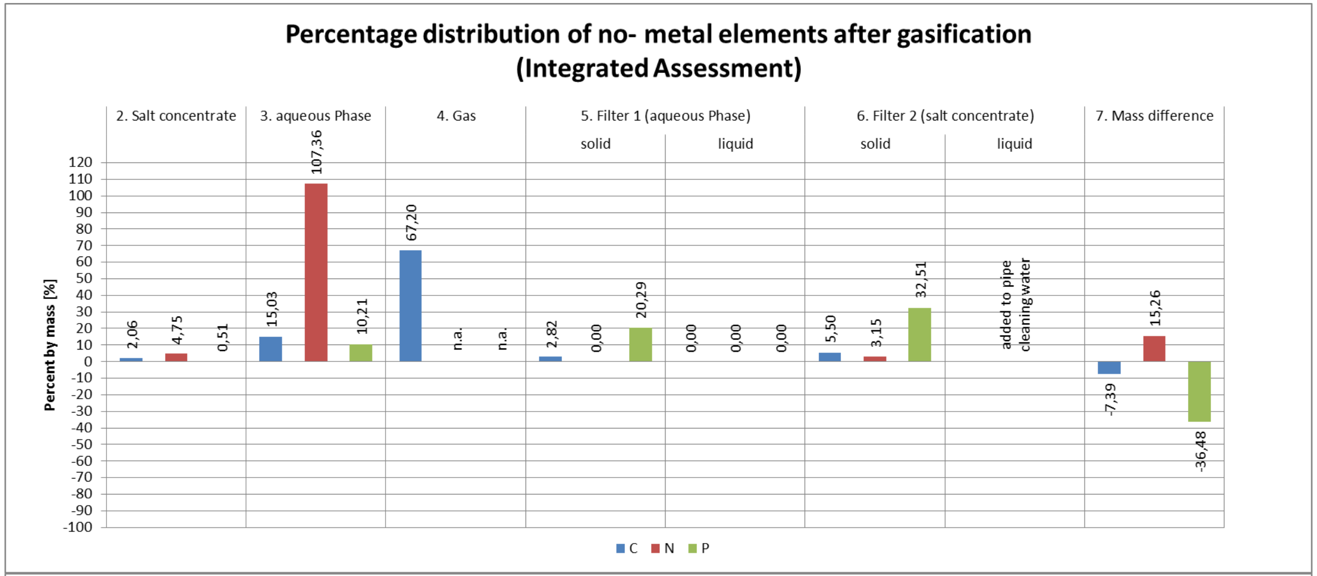
Filter1 – aqueous phase m = 26 g

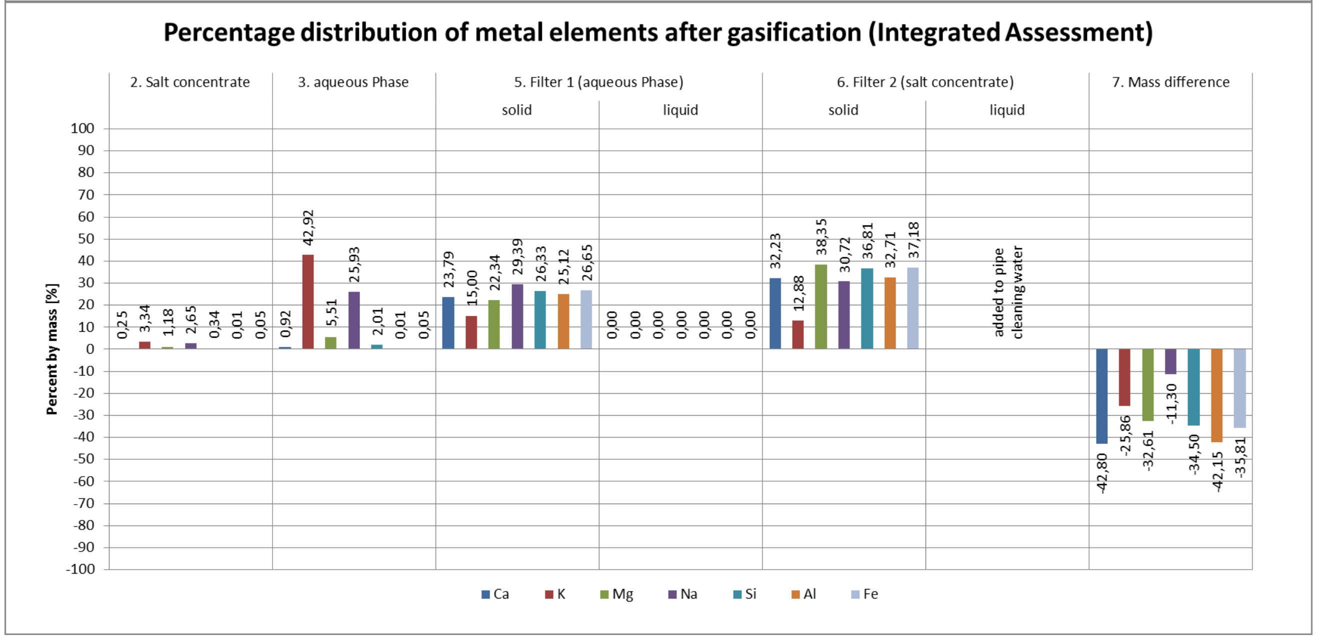
Filter2 – salt concentrate m = 33 g

Method: ICP-AAS, except N and except the cursive values in brackets, which were measured by EDX EDX: O-values






Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
2,06
4,75
0,51
15,03
107,36
10,21
67,20
n.a.
n.a.
2,82
0,00
20,29
0,00
0,00
0,00
5,50
3,15
32,51
added to pipe cleaning water
-7,39
15,26
-36,48
C
N
P


Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
6. Filter 2 (salt concentrate)
7. Mass difference
solid
liquid
Percent by mass [%]
0,25
3,34
1,18
2,65
0,34
0,01
0,05
0,92
42,92
5,51
25,93
2,01
0,01
0,05
23,79
15,00
22,34
29,39
26,33
25,12
26,65
0,00
0,00
0,00
0,00
0,00
0,00
0,00
32,23
12,88
38,35
30,72
36,81
32,71
37,18
added to pipe cleaning water
-42,80
-25,86
-32,61
-11,30
-34,50
-42,15
-35,81
Ca
K
Mg
Na
Si
Al
Fe

As, Cd, Hg and Pb could not be detected in the product streams. As and Cd were also not detectable in the feed. In the feed used some 0,0002 g Hg and 0,04 g Pb were detected.

Cu-balance		
	[g Cu]	[%]
Cu- amount feed:	0,211	100
Cu- amount outlet + cold water cleaning phase + V41:	0	0
Cu- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0,0572	27,1
filter F01 - liquid:	0	0
filter F02 - salt separation system / solid:	0,0759	35,9
filter F02 - salt separation system / liquid:	0	0
Cu- amount result:	0,133	63
deficit:	-0,0781	-37,0
Zn-balance		
	[g Zn]	[%]
Zn- amount feed:	0,344	100
Zn- amount outlet + cold water cleaning phase + V41:	0	0
Zn- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0,0936	27,2
filter F01 - liquid:	0	0
filter F02 - salt separation system / solid:	0,122	35,5
filter F02 - salt separation system / liquid:	0	0
Zn- amount result:	0,216	62,8
deficit:	-0,128	-37,2

2.4.16 Experiment STOWA 16

In Experiment STOWA 16 the dry matter content of the feed was increased to 15 wt.-%. During the tests with sewage sludge from Lelystad with such a high dry matter content plugging occurred, but this test ran stable.

Experiment conditions		
Dry matter content	15,00	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	653	°C
pressure	280	bar
real residence time	2,75	min
real Feed mass flow	423	g/h

Sewage sludge		
time	2,00	h
Total sludge fed	846	g
C- mass in the total feed*	51,01	g
Total mass effluent	547	g
Total mass salt brine	90,5	g
TOC-Conc.		
Effluent sample	2,39	mg/g
Salt conc. sample (Position 2)	1,09	mg/g
Gas production	37,55	Nl/h
Conversion of gasification (TC)	75,9	%
TOC-conversion**	96,5	%
Mass-balance	106,3	%
C-balance (TC)	81,6	%
C-deficit (TC)	-6,7	g
Gas composition		
H_2	13,76	Vol %
CO	0,44	Vol %
CO_2	41,24	Vol %
CH_4	32,86	Vol %
C_2H_4	0,46	Vol %
C_2H_6	10,85	Vol %
C_3H_6	0,10	Vol %
C_3H_8	0,29	Vol %

* TIC in the feed not detectable
** without the C from $KHCO_3$

STOWA 16								Filter solid	
	Feed analysis		**aq. Effluent analysis during experiment (Position 3)**				**salt concentrate analysis (Position 2)**	**Filter1 (Position 5)**	**Filter2 (Position 6)**
Parameter	Concentration dry matter	Concentration diluted sludge*	after 5 hours	after 6 hours	after 7 hours	after experiment	after experiment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	
TC	402	60300	9268	9034	8808	6678	3377	315	169 *(279)*
TIC	n.d.	n.d.	6575	5990	6368	4571	154	n.d.	n.d.
TOC (C)	402	60300	2693	2044	2440	2006	3223	315	169 *(279)*
H	60,6	9090	-	-	-	-	-	-	-
N	47,4	7110	-	-	-	-	-	19,3	10,7
O	-	-	-	-	-	-	-	349	386
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NH4	-	-	11560	12680	13000	7120	703	-	-
TNb	-	-	7798	6806	7487	5678	923	-	-
P	21,8	3270	307	314	327	194	27,1	60,7	76,4 *(77,3)*
PO4	-	-	198	207	246	181	56,0	-	-
S	8,7	1305	129	150	158	95,5	152	12,9	n.d.
SO4	-	-	155	144	130	71,0	136	-	-
Ca	16,3	2445	20,0	23,5	24,1	16,2	14,1	53,6	55,4
K	2,96	444	922	888	901	701	300	45	32,2
Mg	2,86	429	0,40	0,81	0,75	1,63	12	8,6	11,7
Na	0,84	126	24,4	26,4	26,7	14,8	6	3,1	3,1
Si	35,3	5295	47,7	48,5	50,1	53,9	46,4	131	164
Al	12,2	1830	n.d.	n.d.	n.d.	n.d.	n.d.	40,3	40,8
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,59	88,5	n.d.	n.d.	n.d.	n.d.	n.d.	2,2	2,2
Pb	0,111	16,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,96	144	n.d.	n.d.	n.d.	n.d.	n.d.	3,6	3,6
Cr	0,042	6,3	n.d.	n.d.	n.d.	n.d.	n.d.	0,17	0,18
Fe	20,9	3135	0,72	0,31	0,20	0,15	14,0	81	82,4
Mo	0,015	2,25	n.d.	n.d.	n.d.	n.d.	n.d.	0,64	n.d.
Ni	0,024	3,6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cl	2,83	425	113	112	97	124	74,6	0,997	0,298
Hg	0,00063	0,0945	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	575	583	580	415	51	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Oijen sample 18.07.14

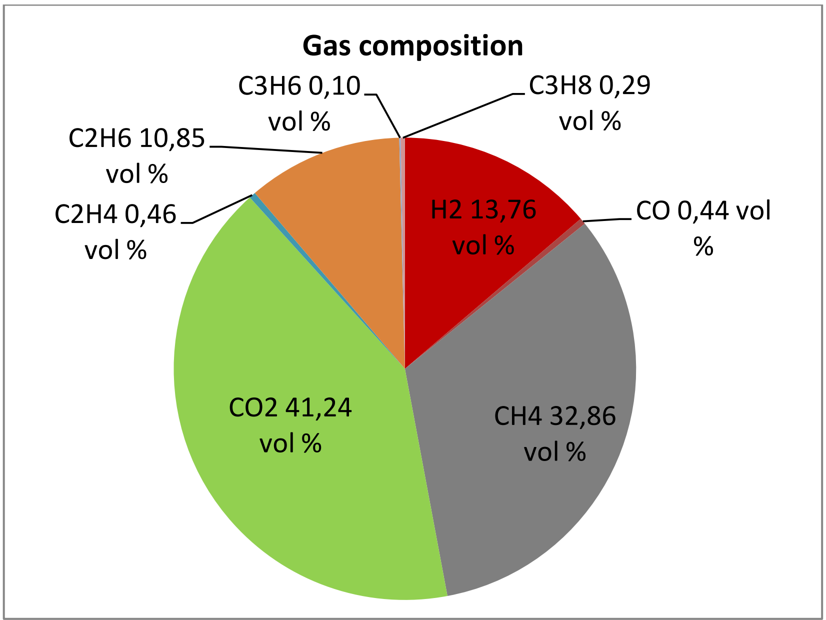
Feed			
HHV:		16504	J/g
Ash content	by 550°C:	22,45	%
	by 815°C:	22,17	%
	by 1000°C:	21,82	%

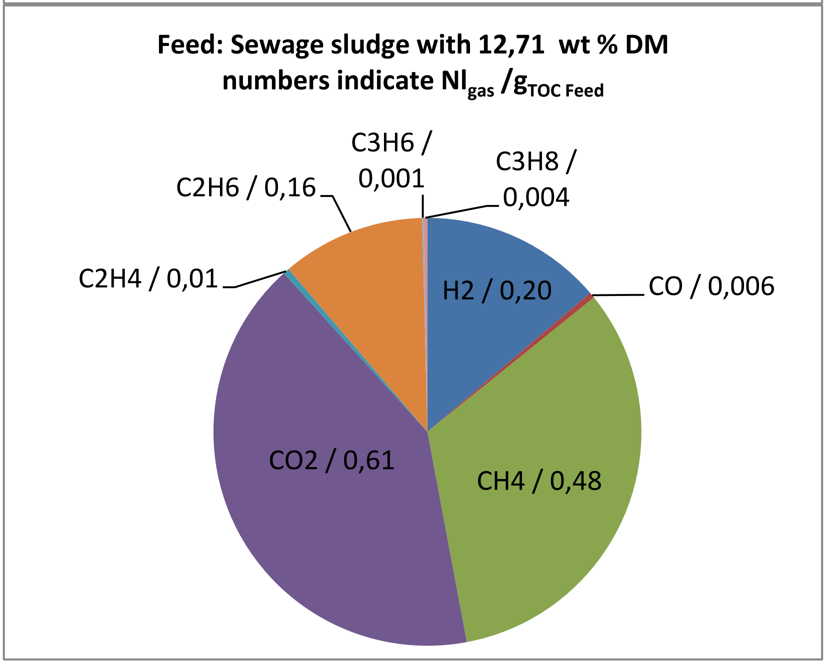
<u>Mass of solids:</u>

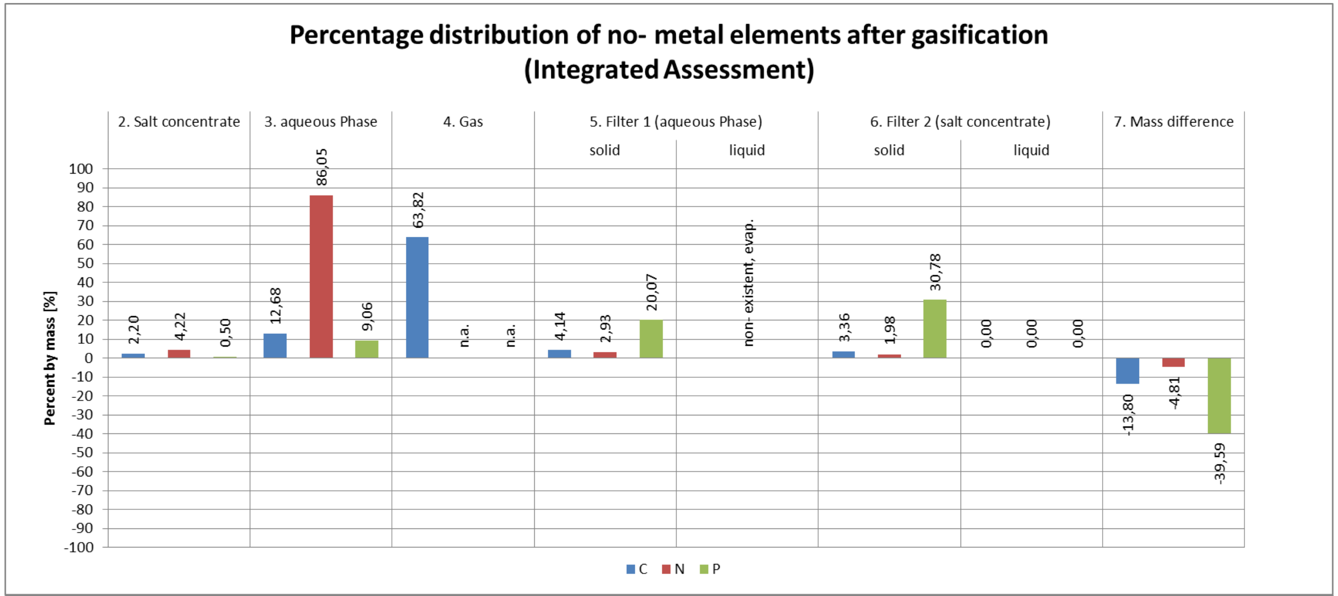
Filter1 – aqueous phase m = 32 g

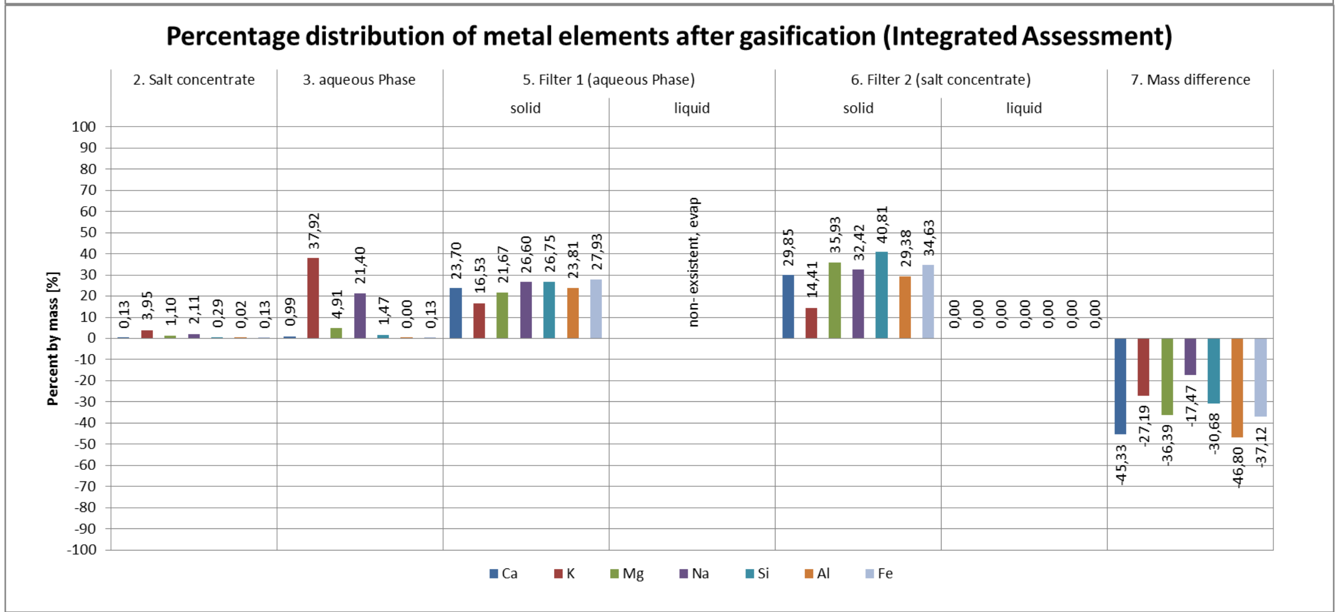
Filter2 – salt concentrate m = 39 g

Method: ICP-AAS, except N and except the cursive values in brackets, which were measured by EDX EDX: O-values






Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
solid
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
2,20
4,22
0,50
12,68
86,05
9,06
63,82
n.a.
n.a.
4,14
2,93
20,07
non- existent, evap.
3,36
1,98
30,78
0,00
0,00
0,00
-13,80
-4,81
-39,59
C
N
P


Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
solid
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
0,13
3,95
1,10
2,11
0,29
0,02
0,13
0,99
37,92
4,91
21,40
1,47
0,00
0,13
23,70
16,53
21,67
26,60
26,75
23,81
27,93
non- exsistent, evap
29,85
14,41
35,93
32,42
40,81
29,38
34,63
0,00
0,00
0,00
0,00
0,00
0,00
0,00
-45,33
-27,19
-36,39
-17,47
-30,68
-46,80
-37,12
Ca
K
Mg
Na
Si
Al
Fe

As, Cd, Hg and Pb could not be detected in the product streams. As and Cd were also not detectable in the feed. In the feed used some 0,0003 g Hg and 0,049 g Pb were detected.

Cu-balance		
	[g Cu]	[%]
Cu- amount feed:	0,262	100
Cu- amount outlet + cold water cleaning phase + V41:	0	0
Cu- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0,0704	26,9
filter F01 - liquid:	0	0
filter F02 - salt separation system / solid:	0,0858	32,8
filter F02 - salt separation system / liquid:	0	0
Cu- amount result:	0,156	59,6
deficit:	-0,106	-40,4
Zn-balance		
	[g Zn]	[%]
Zn- amount feed:	0,426	100
Zn- amount outlet + cold water cleaning phase + V41:	0	0
Zn- amount salt concentrate + cold water cleaning phase + V33:	0	0
filter F01 - solid:	0,115	27,0
filter F01 - liquid:	0	0
filter F02 - salt separation system / solid:	0,14	32,9
filter F02 - salt separation system / liquid:	0	0
Zn- amount result:	0,256	60,0
deficit:	-0,171	-40,0

2.4.17 Experiment STOWA 17

For experiment STOWA 17 the dry matter content was increased to the highest value (17,37 wt.-%) of all experiments. The feed flow rate was lowered to obtain a residence time of 5,43 min. The experiment proceeded stable during the feed phase. After 6 h a blockage of the flow rate occurred within the tee and the line between the preheater and the reactor. For this reason the cleaning phase was then started and ran irregular. The particles which were responsible for plugging are not analysed.

Experiment conditions		
Dry matter content	17,37	%
K^+-conc. ($KHCO_3$)	2500	mg/l
real Temperature	648	°C
pressure	280	bar
real residence time	5,43	min
real Feed mass flow	210,5	g/h

Sewage sludge		
time	2,00	h
Total sludge fed	421	g
C- mass in the total feed*	31,01	g
Total mass effluent	147	g
Total mass salt brine	99	g
TOC-Conc.		
Effluent sample	0,80	mg/g
Salt conc. sample (Position 2)	4,58	mg/g
Gas production	17,03	Nl/h
Conversion of gasification (TC)	48,6	%
TOC-conversion**	97,2	%
Mass-balance	115,9	%
C-balance (TC)	58,3	%
C-deficit (TC)	-13,1	g
Gas composition		
H_2	25,74	Vol %
CO	0,46	Vol %
CO_2	29,69	Vol %
CH_4	35,94	Vol %
C_2H_4	0,10	Vol %
C_2H_6	8,01	Vol %
C_3H_6	0,01	Vol %
C_3H_8	0,06	Vol %

* TIC in the feed not detectable
** without the C from $KHCO_3$

STOWA 17								Filter solid	
	Feed analysis		aq. Effluent analysis during experiment (Position 3)				salt concentrate analysis (Position 2)	Filter1 (Position 5)	Filter2 (Position 6)
Parameter	Concentra-tion dry matter	Concentra-tion diluted sludge*	after 4 hours	after 5 hours	after 6 hours	after experiment	after experiment	Concentration dry matter	
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	
TC	424	73649	6539	7595	8380	5336	8761	358	276
TIC	n.d.	n.d.	5855	6754	7504	4438	299	n.d.	n.d.
TOC (C)	424	73649	684	841	876	898	8461	358	276
H	64,3	11169	-	-	-	-	-	-	-
N	44,3	7695	-	-	-	-	-	19,7	17,2
O	-	-	-	-	-	-	-	-	-
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	-	-
NH4	-	-	9140	10600	11400	5970	857	-	-
TNb	-	-	5855	6528	6754	4605	1716	-	-
P	20,40	3543	54,3	65,7	78,9	44,7	50,1	55,7	84,5
PO4	-	-	188	213	218	75,0	147	-	-
S	6,68	1160	16,0	22,1	29,4	18,7	198	147	125
SO4	-	-	41,2	59,6	74,7	47,1	211	-	-
Ca	13,9	2414	5,10	3,74	3,06	5,55	21,6	46,4	58
K	2,82	490	201	400	550	247	729	35,7	34,6
Mg	2,61	453	4,00	5,70	7,00	4,24	25,2	7,4	10,9
Na	0,67	117	2,82	5,31	7,16	4,32	10,2	2,35	2,6
Si	15,79	2743	20,2	27,2	33,1	23,7	53,4	148	114
Al	9,31	1617	0,20	0,17	0,15	n.d.	n.d.	43,7	39
As	n.d	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	0,45	77,3	n.d.	n.d.	n.d.	n.d.	n.d.	2	2,1
Pb	0,05	8,23	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	0,48	83,3	n.d.	n.d.	n.d.	n.d.	0,06	3,1	2,3
Cr	0,02	4,21	n.d.	n.d.	n.d.	n.d.	n.d.	0,29	0,26
Fe	18,5	3213	1,53	0,85	0,83	1,3	2,66	54,8	80
Mo	0,01	1,32	n.d.	n.d.	n.d.	n.d.	n.d.	0,29	n.d.
Ni	0,01	2,49	n.d.	n.d.	n.d.	n.d.	n.d.	4,4	1,7
Cl	2,11	367	93,6	153	184	75	147	0,266	0,421
Hg	-	-	0,0	0,0	0,0	0,0	0,0	n.d.	n.d.
Phenolindex	-	-	265	354	358	196	93	-	-

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend	
n.d.	not detectable or signal interference

Additional analytical data: Sewage sludge from Oijen sample 16.09.14

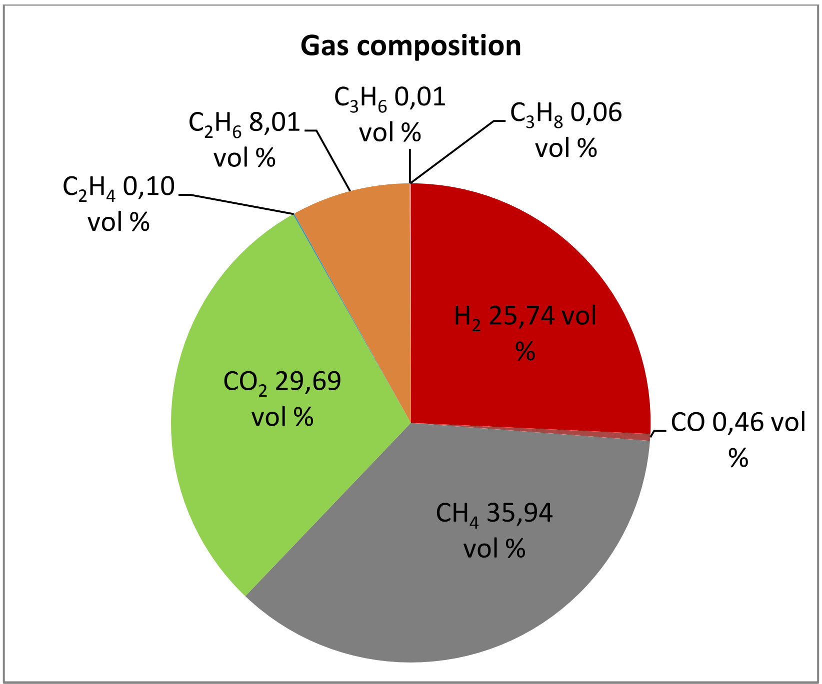
Feed			
HHV:		18105	J/g
Ash content	by 550°C:	15,91	%
	by 815°C:	15,62	%
	by 1000°C:	14,97	%

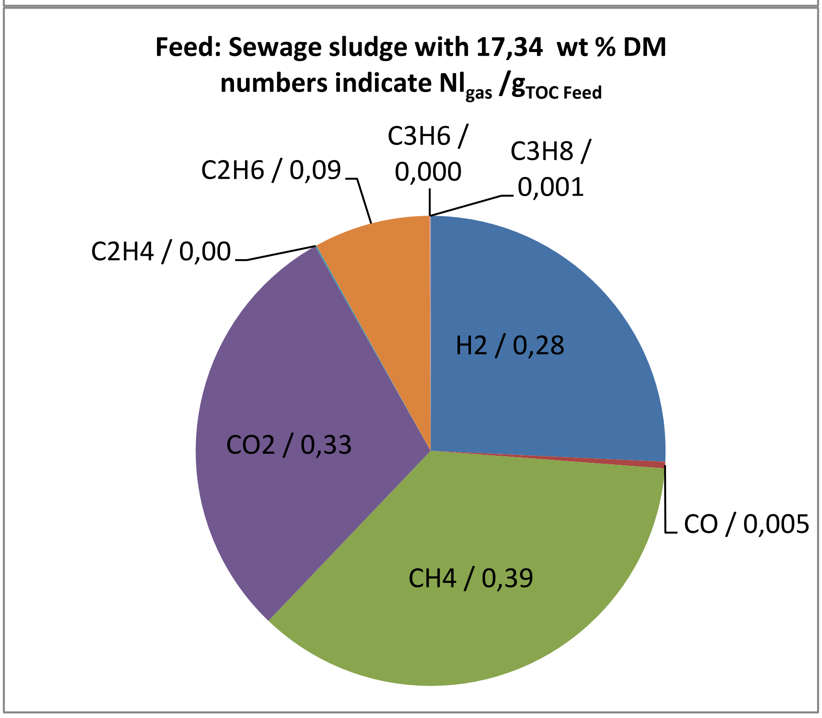
<u>Mass of solids:</u>

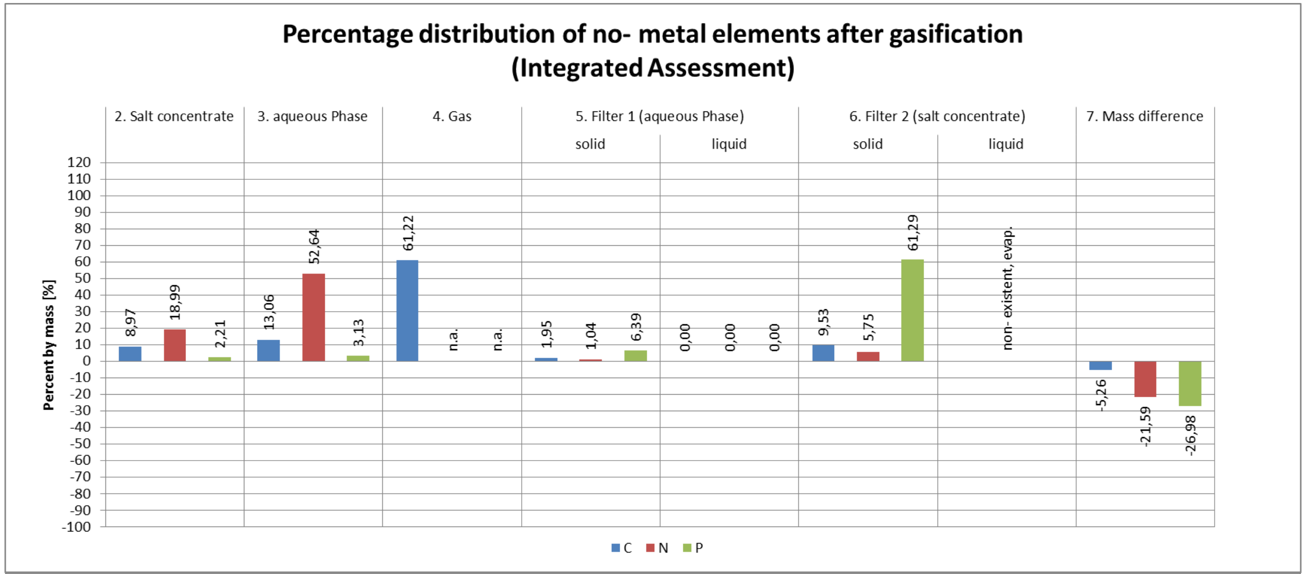
Filter1 – aqueous phase m = 5,2 g

Filter2 – salt concentrate m = 32,9 g

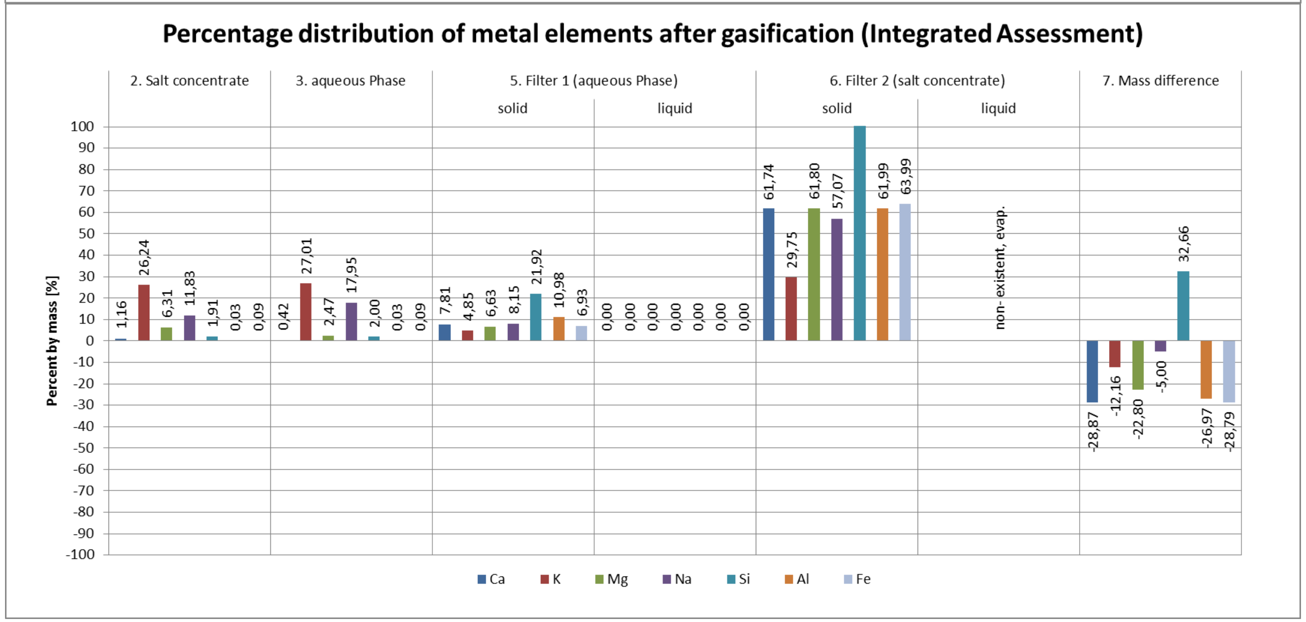
Method: ICP (except N); EDX: C and O-values







Percentage distribution of no- metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
4. Gas
5. Filter 1 (aqueous Phase)
solid
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
8,97
18,99
2,21
13,06
52,64
3,13
61,22
n.a.
n.a.
1,95
1,04
6,39
0,00
0,00
0,00
9,53
5,75
61,29
non- existent, evap.
-5,26
-21,59
-26,98
C
N
P



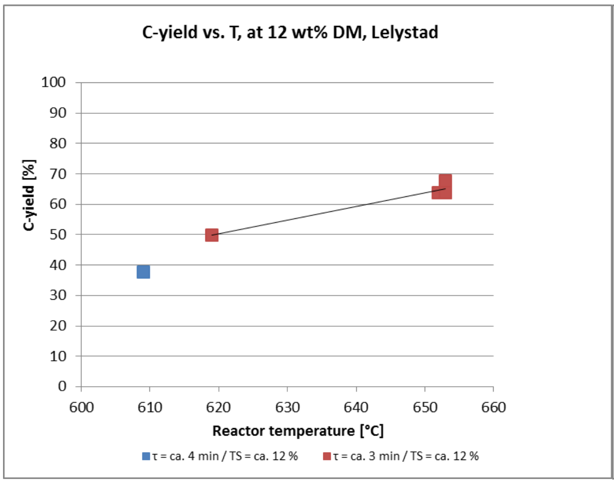
Percentage distribution of metal elements after gasification (Integrated Assessment)
2. Salt concentrate
3. aqueous Phase
5. Filter 1 (aqueous Phase)
solid
liquid
6. Filter 2 (salt concentrate)
solid
liquid
7. Mass difference
Percent by mass [%]
1,16
26,24
6,31
11,83
1,91
0,03
0,09
0,42
27,01
2,47
17,95
2,00
0,03
0,09
7,81
4,85
6,63
8,15
21,92
10,98
6,93
0,00
0,00
0,00
0,00
0,00
0,00
0,00
61,74
29,75
61,80
57,07
61,99
63,99
non- existent, evap.
-28,87
-12,16
-22,80
-5,00
32,66
-26,97
-28,79
Ca
K
Mg
Na
Si
Al
Fe

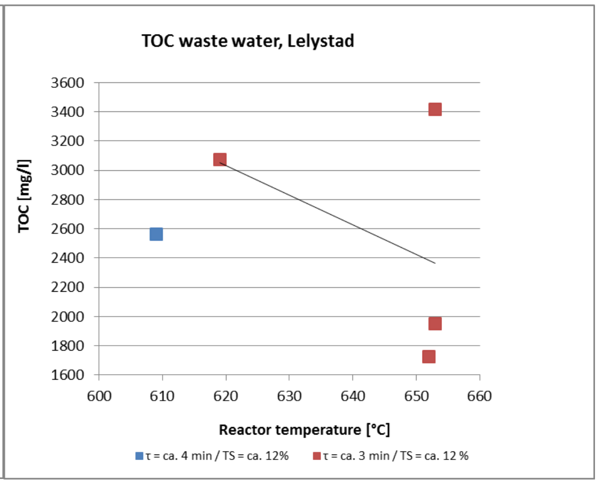
3 Influence of the process parameters on the products

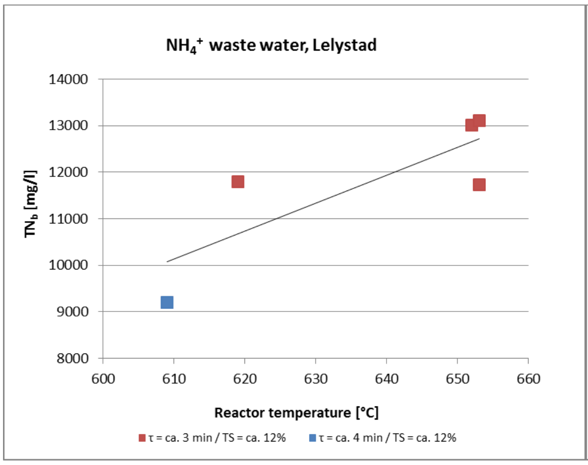
The experiments showed that the reaction temperature has the greatest influence on the gasification yield. Generally with sewage sludge from Oijen higher gasification yields were reached than with sewage sludge from Lelystad. In the following the influence of the process parameters on the products will be addressed.

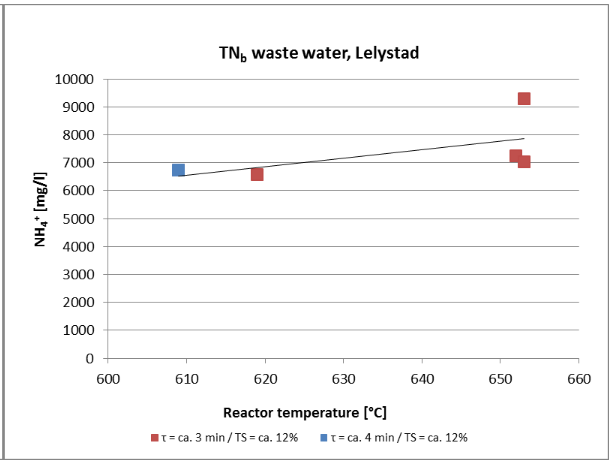
3.1 Influence of the reaction temperature

The C-gasification yield increases with the reactor temperature. Correspondingly there is a trend of decreasing TOC content in the waste water with the reactor temperature while the TN_b and NH_4^+ values increase. The variation of the different values is high. Due to the low number of experiments it is not possible to define runaway values.





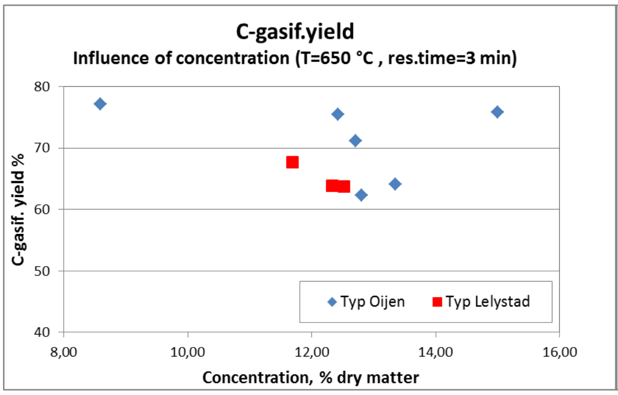


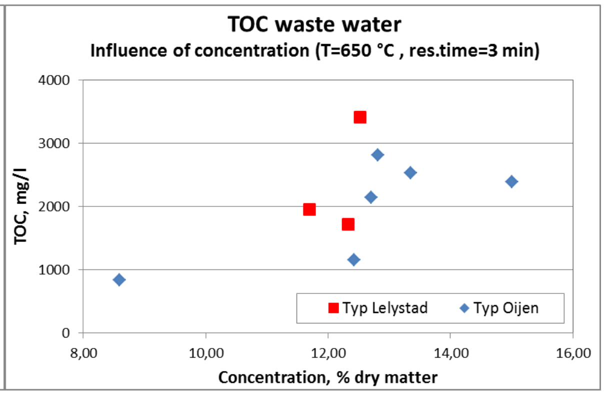


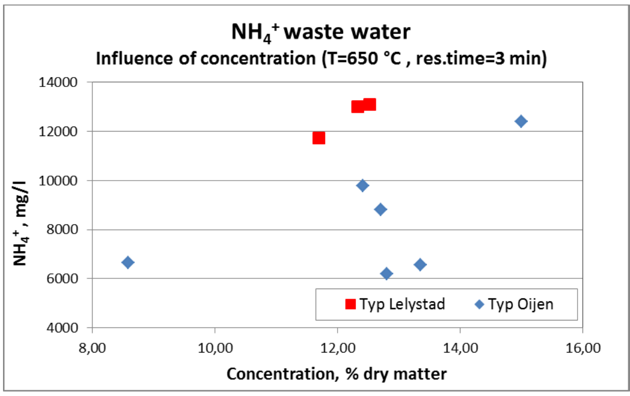
The influence of the reaction temperature on the gasification during the experiments with the sludge from Oijen has not been studied. All experiments with this sludge were performed at a reaction temperature of 650 °C. Notice: the N-concentration in the salt concentrate is low.

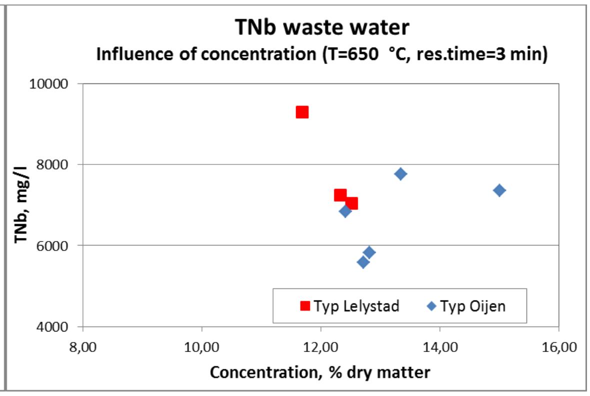
3.2 *Influence of the concentration of the feed*

The scattering of the results in the diagrams below is too high to make a statement about the influence of the concentration of the feed. Probably many parameters influence the results. The TOC content increases over proportional compared to the dry matter concentration. This is an indication for lower conversion rate at increased dry matter content. The TNb is always lower than NH_4^+. The reason is analytical error due to not known inteferences from other species in the samples.









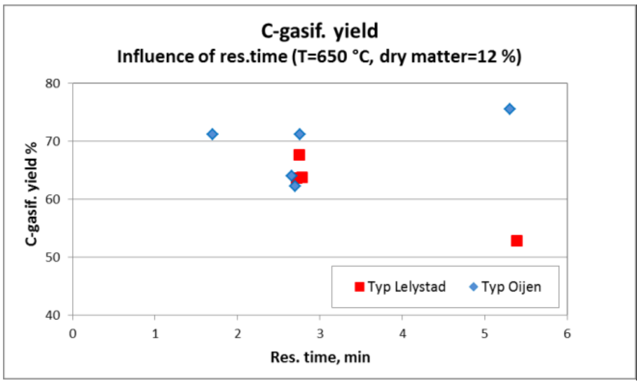
In Table 6 the C-balances at a reactor temperature of 650 °C and a mean residence time of 3 minutes are listed. The values show that the dry matter content does not influence the C-balance. This is an indication of no significantly increased shoot and tar formation at higher dry matter concentrations.

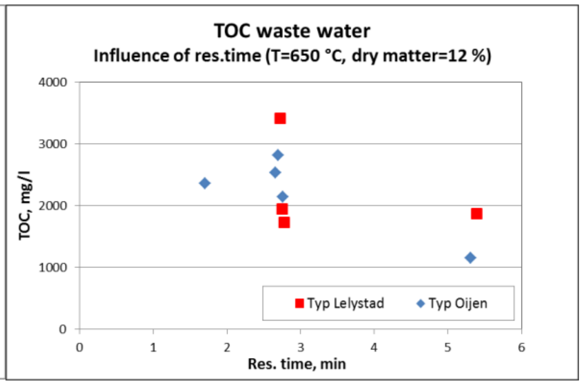
Table 16: C-balance at 650 °C and 3 min mean residence time.

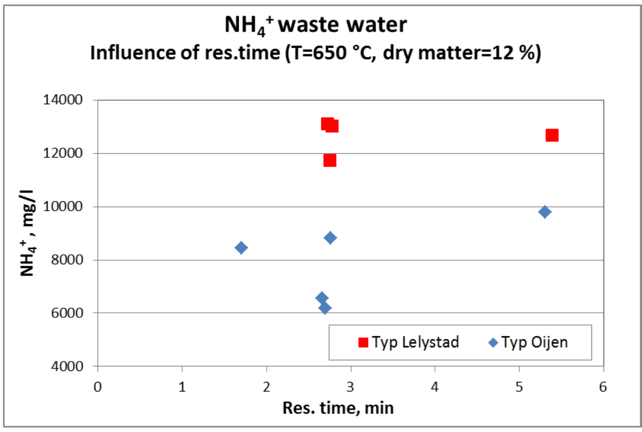
		TS %	C- Balance [%]
Typ Lelystad	5	12,52	80,74
	7	12,33	77,7
	9	11,69	80,49
Typ Oijen	10	12,71	81,83
	12	12,42	85,84
	13	8,59	86,87
	14	12,81	73,87
	15	13,35	77,31
	16	15,00	81,59

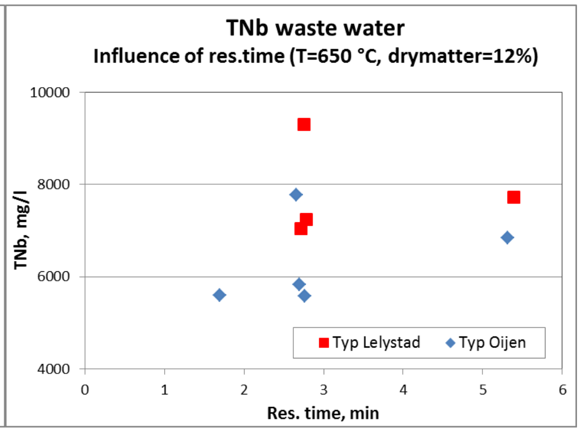
3.3 Influence of the residence time

The scattering of the results in the diagrams below is again too high to make a statement about the influence of the residence time. Probably many other parameters influence the results







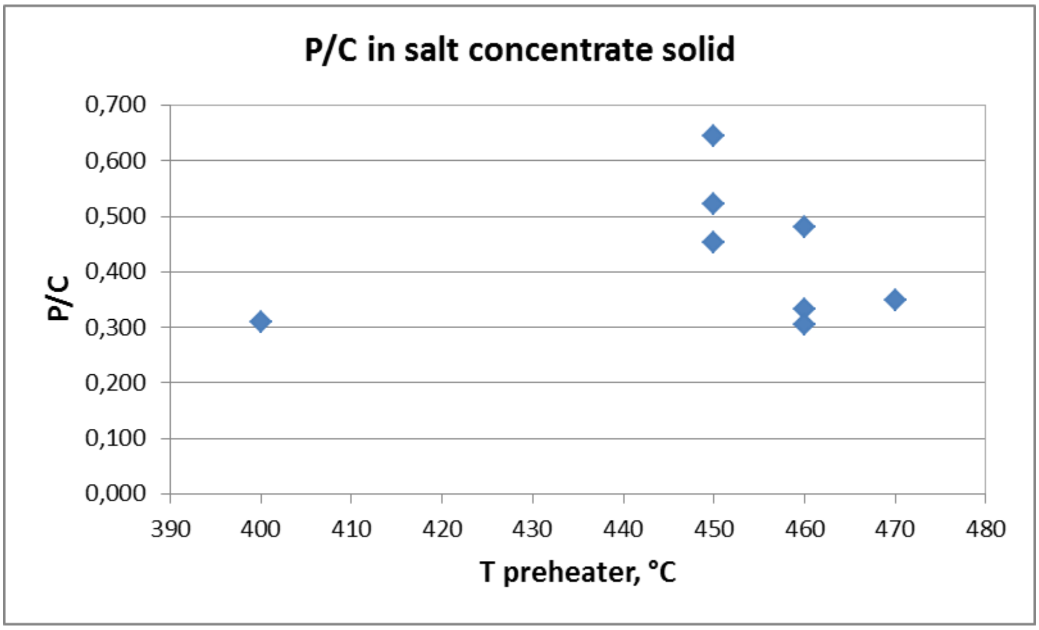


3.4 Influence of the preheater temperature on the salt separation

In the following Table 17 the ratio of phosphorus and carbon found in the solid residue of filter 2 of the salt separation line is listed. However no clear tendency can be observed.

Table 17: P/C ratio in the solid of filter 2 (salt concentrate) (method: P-ICP; C-DIN EN 13137)

Experiment	10	11	12	13	14	15	16	17
P, mg/g	76,3	86,5	88,4	70,9	69,4	76,9	76,4	84,5
C, mg/g	219	180	137	136	208	248	169	276
P/C	0,348	0,481	0,645	0,521	0,334	0,310	0,452	0,306
T preheater	470	460	450	450	460	400	450	460



P/C in salt concentrate solid
0,700
0,600
0,500
0,400
0,300
0,200
0,100
0,000
P/C
390
400
410
420
430
440
450
460
470
480
T preheater, °C

4 Pilot scale experiment KS1

In the following the run and the results of the pilot-scale test in the VERENA facility are presented.

4.1 Process flow sheet of the pilot plant

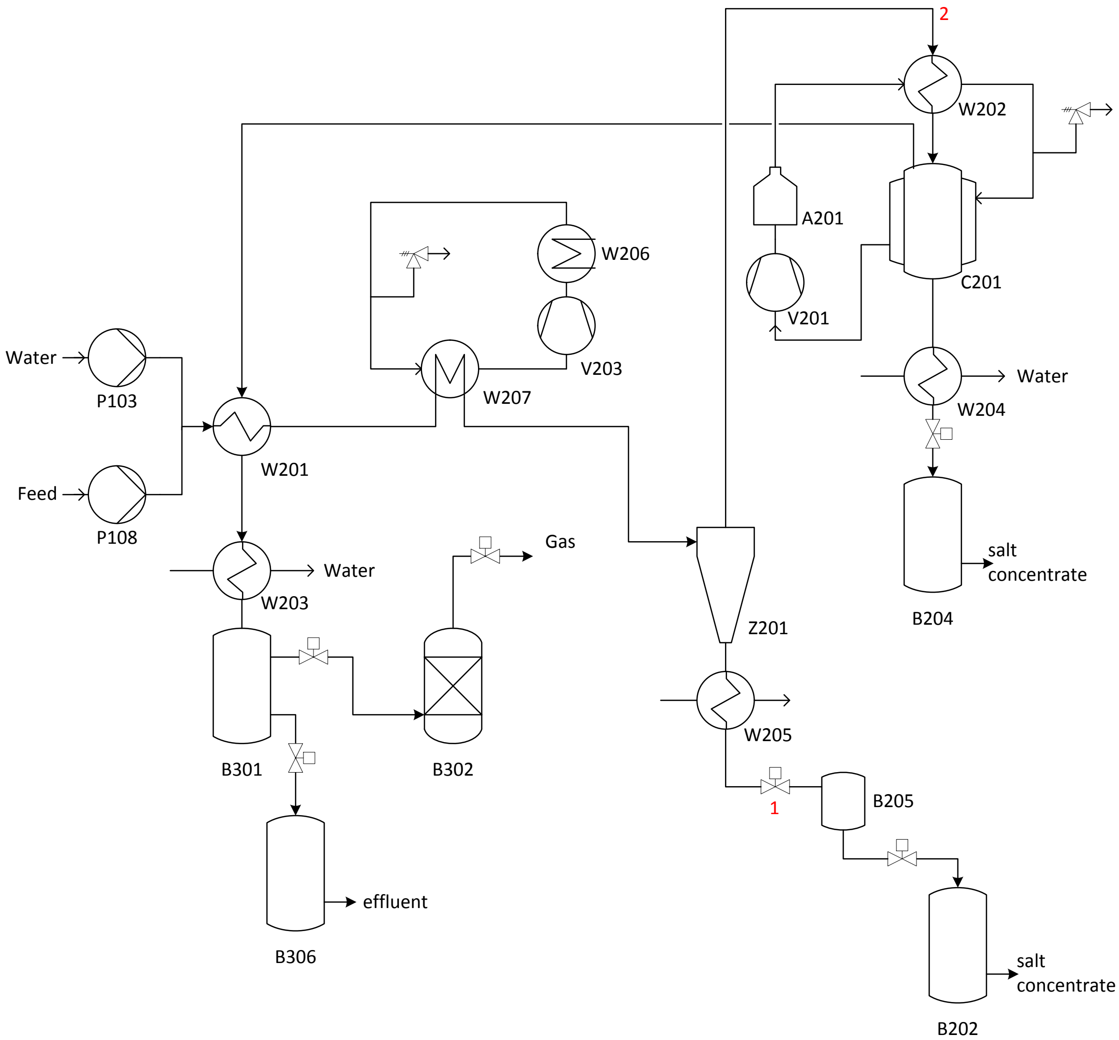


Figure 8: Process flow sheet of the VERENA plant (the buffer tank B205 was first implemented for the second pilot-scale experiment)

P103: diaphragm pump with 2 pump heads

P108: hose diaphragm pump with 1 pump head, a lower frequency and a bigger valve diameter (28 mm) than P103 (valve diameter: 10 mm)

A201: propane gas burner

B202: collection vessel for the salt concentrate (cyclone)

B204: collection vessel for the salt concentrate (sump reactor)

B205: buffer tank to realize a second pressure step (first implemented after experiment KS1)
C201: reactor (slim vessel)
V201: blower for the recirculation of the flue gas of the heating system of the reactor and the main preheater
V203: blower for the recirculation of the nitrogen gas of the heating system of the salt separation system
W201: double-pipe heat exchanger
W202: main preheater
W203: cooler
W204: cooler for the salt concentrate from the sump of the reactor
W205: cooler for the salt concentrate from the sump of the cyclone
W206: electric heater which heats up nitrogen gas in a closed loop
W207: preheater of the salt separation system
Z201: cyclone, attached to the bottom of the preheater W207
B301: separation vessel
B302: separation vessel equipped with an integrated CO_2 scrubber
B306: vessel for the effluent of the process

4.2 Pretreatment

The sewage sludge from Oijen was delivered in 15 steel vessels which were filled with dewatered sludge cake and centrate liquid from the dewatering process. The content of the barrels is shown in Table 39.

Table 18: Amount of dewatered sludge cake and centrate liquid in the delivered vessels.

nr.	dewatered sludge cake (kg)	Centrate liquid (kg)	Dry matter % in vessel
1	120,8	80,4	13,9
2	120	80,4	13,9
3	122	80,2	14,0
4	120,4	80,2	13,9
5	121	79,8	14,0
6	120,8	80	14,0
7	121,2	80	14,0
8	119,8	80,4	13,9
9	121,4	80,4	14,0
10	121	80	14,0
11	140	60	16,2
12	141,6	60,2	16,3
13	140,2	60,2	16,2
14	140,2	60	16,2
15	139,8	60,2	16,2

The content of every vessel was filled into the tank of a screwpump as shown in Table 9a). Parallel to each barrel 5,5 L KHCO3 solution were added, which results in a concentration of 2500 mg K+/L since potassium salts catalyse the gasification reaction (K+ is added at a concentration of 2500 mg/L sludge additionally to the original K value of the sludge). The sludge with the KHCO3 solution was recirculated for homogenization (see Table 9a)). Subsequently the preconditioned sewage sludge was conveyed into an IBC (see Table 9b)) and homogenized again.

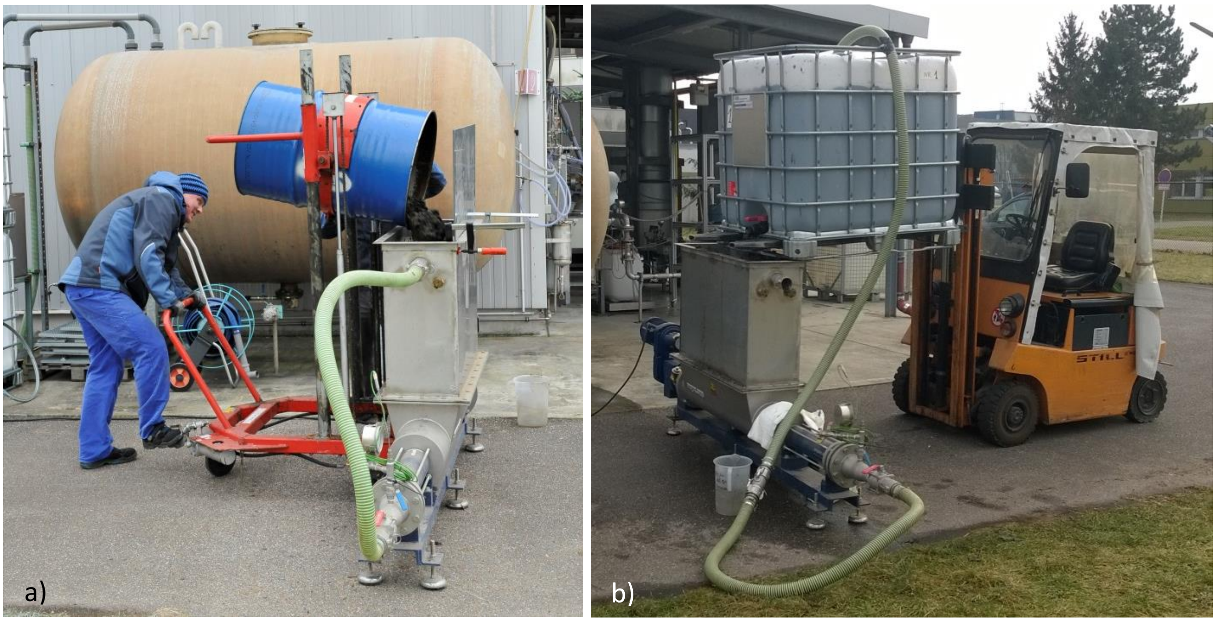


Figure 9: Pretreatment of the feed.

The sludge of overall 4 IBCs was than pumped through a colloid mill for better homogenization. This treatment did not change the macroscopic appearance of the sludge. Furthermore during this procedure 70 kg water were added each to IBC No. 1 and IBC No. 3 while part of the content of IBC No. 1 was transferred in IBC No. 4 after dilution. In Table 40 it is listed in which of the 4 IBCs which vessel was filled. Only sewage sludge of IBC No. 1 was used for experiment KS1, due to premature end of the experiment.

Table 19: Assignment of the barrels to the IBCs.

IBC No.	vessel No.	Dry matter % in IBC
1	6, 7, 8, 9, 10	11,8*
2	1, 2, 3, 4, 5	14,8
3	11, 12, 14, 15	13,4*
4	13, part of IBC No. 1 after dilution	14,7

*determined after dilution with water

After homogenisation and preparation with $KHCO_3$, the feed showed biological activity. This was obvious from the odour, bubbles in the whole mass and a volume expansion of about 20 %.

To determine the dry matter content of the 4 sewage sludge charges respectively ca. 200 g sewage sludge were dried in an oven at 105 °C for 24 h. A double determination of the dry matter of every charge was performed.

4.3 Parameters of the experiment

The pilot plant VERENA was operated at a pressure of 280 bar like the lab-scale apparatus LENA during all 15 experiments with sewage sludge. In Table 41 the parameters of the experiment are summarized. The selection of the experimental parameters is based on the results of the lab scale tests.
The trends of the temperatures in the reactor are depicted in the Appendix.

Table 20: Experimental parameters of the pilot-scale test

No.	Type	Dry Matter of the Feed-sludge	pressure	$T_{Preheater}$ [1] (outlet)	$T_{Reactor}$ [2] (midpoint)	Reactor-residence time	Feed sludge	Feed H_2O
[-]	[-]	[wt-%]	[bar]	[°C]	[°C]	[min]	[kg/h]	[kg/h]
KS-1	Oijen	11,8	280	470	665	1,8	45,3	11,7

[1] $T_{Preheater}$ is the outlet temperature of the preheater W207 of the salt separation system.
[2] $T_{Reactor}$ is the mean midpoint of the triple thermocouple within the reactor C201.

4.4 Detailed description and result of the experiment

The mean residence time is calculated for pure water under operation conditions. This simplification is necessary due to lack of data for sewage sludge under the operation conditions.
The flow rate of the feed results from the predetermined residence time. Thereby the following assumptions (simplifications) are made:

- The sewage sludge consists of dry matter and water, but for simplification it is assumed, that the density of water under operating conditions equates to the density of the sewage sludge under operating conditions.
- The overall density in the reactor is not affected by the occurring gasification reactions.

The residence time of the reactor of the VERENA plant was calculated by the following equation:

$$\tau_{reactor} = \frac{V_{reactor}}{\dot{V}_{Feed}} = \frac{V_{reactor} * \rho_{H2O}}{\dot{m}_{Feed}}$$

In comparison to the calculation of the reactor residence time of the lab-scale test, the reactor was not divided into different temperature zones to calculate residence times of different volume segments.

The balance of the different elements for the experiment is evaluated on two different ways that are time dependent. One balance is formed under constant operating conditions. Most important results of this calculation are the TOC content (TOC = total organic carbon; analysis method: DIN EN 1484) of the waste water, the gas composition and the carbon gasification yield. The second balance is the total balance. This balance is formed over the period of the first sludge input till the very end of sludge feeding. By this calculation a balance of the other elements is also possible.

In the following the equations that were used for evaluation of the pilot-scale tests are specified:

Conversion of gasification

$$Y_{Gas} = \frac{\dot{m}_{TC,Gas}}{\dot{m}_{TC,Feed}} * 100$$

$$\dot{m}_{TC,Gas} = \dot{m}_{TC,PG} + \dot{m}_{TC,PG-GB} + \dot{m}_{TC,LG-effluent} + \dot{m}_{TC,LG-cyclone} + \dot{m}_{TC,LG-reactor}$$

$$\dot{m}_{TC,Feed} = \frac{\dot{m}_{Feed}[\frac{kg}{h}] * DM[\%]}{100} * c_{TC,Feed-DM}[\frac{mg}{g}]/1000[\frac{mg}{g}]$$

In contrast to the lab-scale experiments the phase separation in the pilot plant VERENA takes place under high pressure. Subsequently the lean gas of the separated aqueous phase of vessel B306 is taken into account and also the lean gas of the salt concentrate vessels B202 and B204. The CO_2 solubility in water under ambient conditions is low, about 1.5 g/kg. This is part of the TIC of the waste water.

Y_{Gas}: conversion of the gasification
$\dot{m}_{TC,Gas}$: sum of TC-mass flow of all gas streams
$\dot{m}_{Feed}$: feed mass flow (only sewage sludge)
$c_{TC,Feed-DM}$: concentration of TC in the dry matter of the feed
$\dot{m}_{TC,Feed}$: TC-mass flow of the feed
DM: dry matter content
TC: Total Carbon

C-balance (Y_C):

$$Y_C = \frac{\dot{m}_{TC,out}}{\dot{m}_{TC,Feed}}$$

$$\dot{m}_{TC,feed} = \dot{m}_{Feed-DM} * c_{TC,Feed-DM}$$

$$\dot{m}_{TC,out} = \dot{m}_{TC,reactor} + \dot{m}_{TC,effluent} + \dot{m}_{TC,cyclone-l} + \dot{m}_{TC,cyclone-s} + \dot{m}_{TC,Gas}$$

$\dot{m}_{TC,out}$: TC-mass flow of all output streams
$\dot{m}_{Feed-DM}$: dry matter mass flow of the feed
$\dot{m}_{TC,reactor}$: TC-mass flow of the salt concentrate stream from the sump of the reactor
$\dot{m}_{TC,effluent}$: TC-mass flow of the effluent
$\dot{m}_{TC,cyclone-l}$: TC-mass flow of the sump of the cyclone (liquid)
$\dot{m}_{TC,cyclone-s}$: TC-mass flow of the sump of the cyclone (solid)

Cold gas efficiency (CGE):

$$CGE = \frac{TP_{total\ gas}[W]}{TP_{feed}[W]}$$

$$TP_{total\ gas} = \sum TP_i$$

$$TP_i = \dot{m}_i * HHV_i$$

$$TP_{feed} = \dot{m}_{Feed-DM} * HHV_{DM}$$

$TP_{total\ gas}$: thermal power of total gas
TP_{feed}: thermal power of the feed
TP_i: thermal power of the gas component i
$\dot{m}_i$: mass flow of the gas component i
HHV_i: higher heating value of the gas component i
HHV_{DM}: higher heating value of the dry matter of the feed

TOC-conversion:

$$TOC - conversion = 100 - \frac{\dot{m}_{TOC,out}}{\dot{m}_{TOC,feed}} * 100$$

$$\dot{m}_{TOC,out} = \dot{m}_{TOC,reactor} + \dot{m}_{TOC,effluent} + \dot{m}_{TOC,cyclone-l} + \dot{m}_{TOC,cyclone-s}$$

$$\dot{m}_{TOC,feed} = \dot{m}_{Feed-DM} * c_{TOC,Feed-DM}$$

$\dot{m}_{TOC,out}$: TOC-mass flow of all output streams
$\dot{m}_{TOC,Feed}$: TOC-mass flow of the feed
$\dot{m}_{TOC,reactor}$: TOC-mass flow of the salt concentrate stream from the sump of the reactor
$\dot{m}_{TOC,effluent}$: TOC-mass flow of the effluent
$\dot{m}_{TOC,cyclone-l}$: TOC-mass flow of the sump of the cyclone (liquid)
$\dot{m}_{TOC,cyclone-s}$: TOC-mass flow of the sump of the cyclone (solid)
$c_{TOC,Feed-DM}$: concentration of TOC in the dry matter of the feed

In Table 21 the analysed parameters are listed.

Table 21: Analysed parameters

Feed: dried sludge	Gas composition	Aq. effluent and salt concentrate (aq. phase)
Dry matter content	H_2	
Organic matter content	CO	
Heating value	CO_2	
CHN(O) analysis	CH_4	
TC	C_2H_4	TC
TIC	C_2H_6	TIC
TOC	C_3H_6	TOC
H	C_3H_8	P
P	NH_3	S
S	H_2S	PO4
N	tars	SO4
Ca	Hg	TNb
K		NO_3 and NO_2, NH_4^+
Mg		Ca
Na		K
Si		Mg
Al		Na
As		Si
Cd		Al
Cu		As
Pb		Cd
Zn		Cu
Cr		Pb
Fe		Zn
Mo		Cr
Ni		Fe
Cl		Mo
Hg		Ni
		Cl
		Hg
		Phenol Index

The parameters of Table 21 were analysed analog to the parameters of the lab-scale test (see chapter 4.4). An exception is that the TOC concentration was only calculated via the difference of TC to TIC. The analyses have been performed in three different labs (two in KIT and one by Aquon-NL). Additional measurements of NH_3, H_2S, tars and Hg in the product gas of the pilot scale test were performed.
Gas analysis has been performed in three KIT labs and additionally the Hg measurement has been performed by Tauw (NL). For the analysis of NH_3 and H_2S in the gaseous phase sampling bags -made of aluminium- were filled with the product gas which was analysed offline by Micro-GC. The Hg analysis of the product gas was measured by the method NEN-EN 13211: 2001. This measurement was performed external by Tauw B.V.. To analyse tars (benzene, toluene, naphthalene) in the gas phase a cold trap was installed to transfer the tars into a mixture of water and ethylene glycol. In the forefront of the experiment the gas velocity, temperature, solvent and the time for condensing out was varied to optimize this method. Based on these tests 1,5 NL gas were transferred through 20 mL H_2O:ethylene glycol (9:1) in 30 minutes. The resulting mixture was analysed thereafter by Headspace GC-MS.

4.4.1 Progress of experiment KS1

9th February

To carry out a pilot scale experiment, the VERENA plant first has to be heated. The campaign started on Monday the 9th February 2015 at 8:00 am. The plant was fed with 59 kg water per hour at a pressure of 250 bar. At first the feed stream was 59 kg/h –instead of the planned 75 kg/h- because the pump P103 which was intended for conveying the water did not work properly. So another water pump P104 was used at the beginning. The temperature of the two heating systems (for the salt separator and for the preheater and the reactor) was increased by 60 °C/h. At 12 o'clock the pump P103 had been repaired and delivered 75 kg water/h into the plant while the pump P104 had been switched off. At 5:25 pm the pressure was increased to 280 bar. To prepare the operation with sewage sludge the IBC No. 1 was brought into the VERENA hall at 10:30 pm and the sludge was pumped in a circle for homogenization.

10th February

At about 6:00 am on Tuesday the lines for the separation of the salts from the sump of the cyclone and from the sump of the reactor were put into operation. After the desired temperatures (470 °C after the salt separation system and mean reactor temperature 660 °C) were reached again the two tanks for the collection of the salt concentrates and the waste water tank were drained. At this point the plant was in operation with water at high temperatures for several hours.

Subsequently at 9:10 am sewage sludge was pumped into the reaction system while the flow of water was reduced (see Figure 10). However the delivery of sewage sludge into the plant had to be stopped after 45 minutes because the flare failed and the hose between the IBC No. 1 and the high pressure pump burst. For about 3 ¼ hours the plant was fed with 70 kg water per hour till the flare could be ignited again and the hose was repaired. During this period of time the line for the separation of salts from the sump of the cyclone had to be taken out of operation for a while because a leakage in this line occurred (see Figure 12). It was not a problem since the system was fed only with water. At 1:00 pm the operation with sewage sludge was restarted but a feeding rate of 75 kg/h was not achievable. This is most probably due to the visible gas bubbles in the feed slurry, which reduced the density of the feed and thus the weight/hour rate. The flow rate is measured by mass flow meters and was all the time correct. Pressure peaks from the sludge pump lead frequent to interruptions of the process. This is due to the high viscosity of the concentrated sludge and the high feeding rate. The amount of water was gradually reduced to a minimum value of 10 kg/h. During the feeding of sewage sludge the line for the removal of the salt concentrate from the sump of the cyclone was generally overloaded. When suddenly a plug appeared this was solved by increasing the opening time of the appropriate pneumatic valve (see Figure 8, position 1). The time interval for opening and closing of this pneumatic valve had to be adjusted several times to compensate variation in the flow rate at the sump of the cyclone. Higher flow rates indicated that the valve was leaking due to erosion by solids. Between 4:16 pm and 4:33 pm the first gas bottle (GF 3) was filled with product gas, while the second gas bottle (GF 12) was filled from 6:35 pm to 7:14 pm (see Figure 11). During operation with sewage sludge, after few hours the pressure in the entrance of the plant became higher (more than 10 bar) than the pressure in the reactor and the exit part of the plant. This was an indication for general fouling. Most of this increase occurred in the main preheater (W207) of the plant. The pressure difference there steadily increased so that the feeding with sewage sludge had to be stopped at 10:00 pm. The plant was flushed with water in order to prevent the plant from total plug. Thereby a pressure peak caused the bursting of a rupture disk at 11:00 pm and the plant had to be cooled down.

<u>After experiment KS1</u>

After cooling down a very persistent plug in the section between the cyclone and the reactor appeared. It took several days of work and very high pressures (600 bar) in order to remove this plug. The working hypothesis of this is that during normal operation fouling appeared in the preheater. After the pressure in the first part of the plant exceeds 300 bar, the rupture disk failed and a reverse very fast flow started. This fast flow removed part of the solids (fouling) covering the inner surface of the pre-heater in the reverce direction and produced the persistent plug.

4.4.2 Experimental data KS1

In the following the operation diagrams (screen shots) of experiment KS1 are shown. In Figure 14 and Figure 15 the in- and out-streams of experiment KS1 are depicted as bar diagrams.

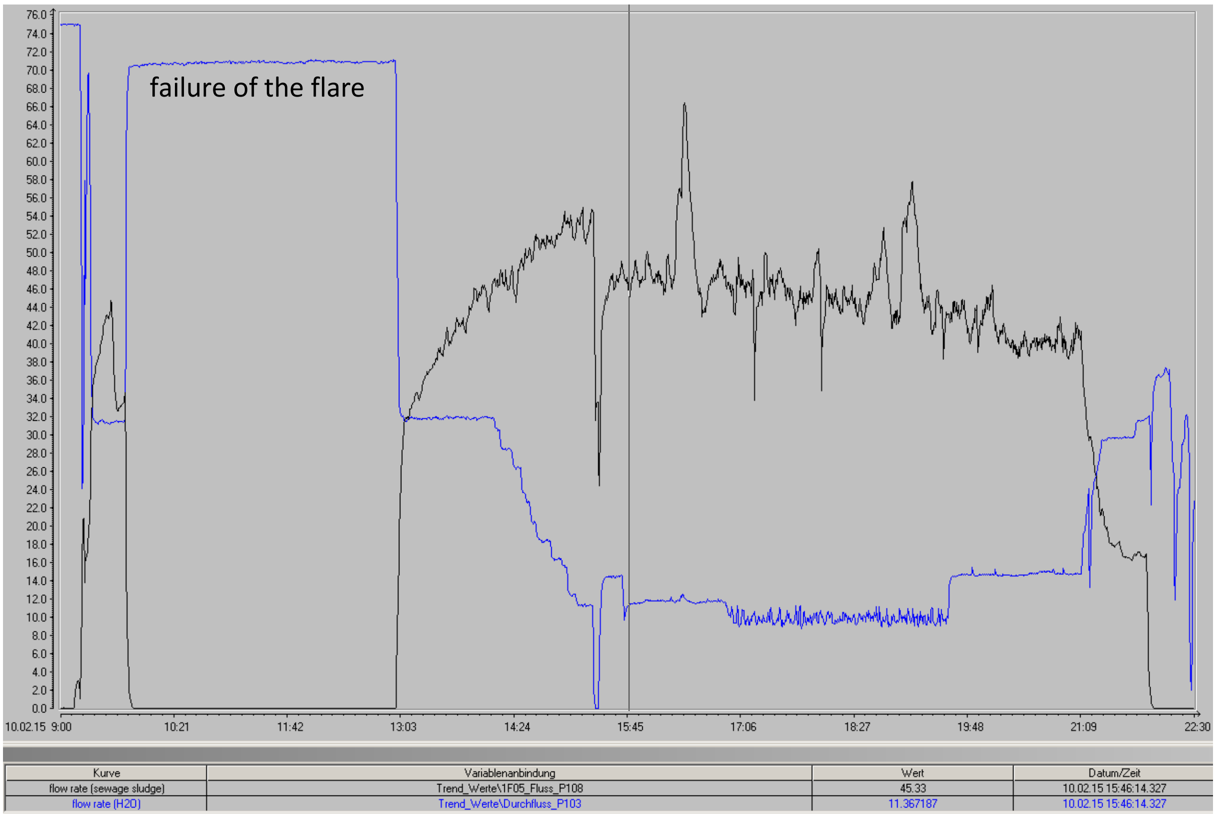


Figure 10: Feed stream of the VERENA test in kg/h.

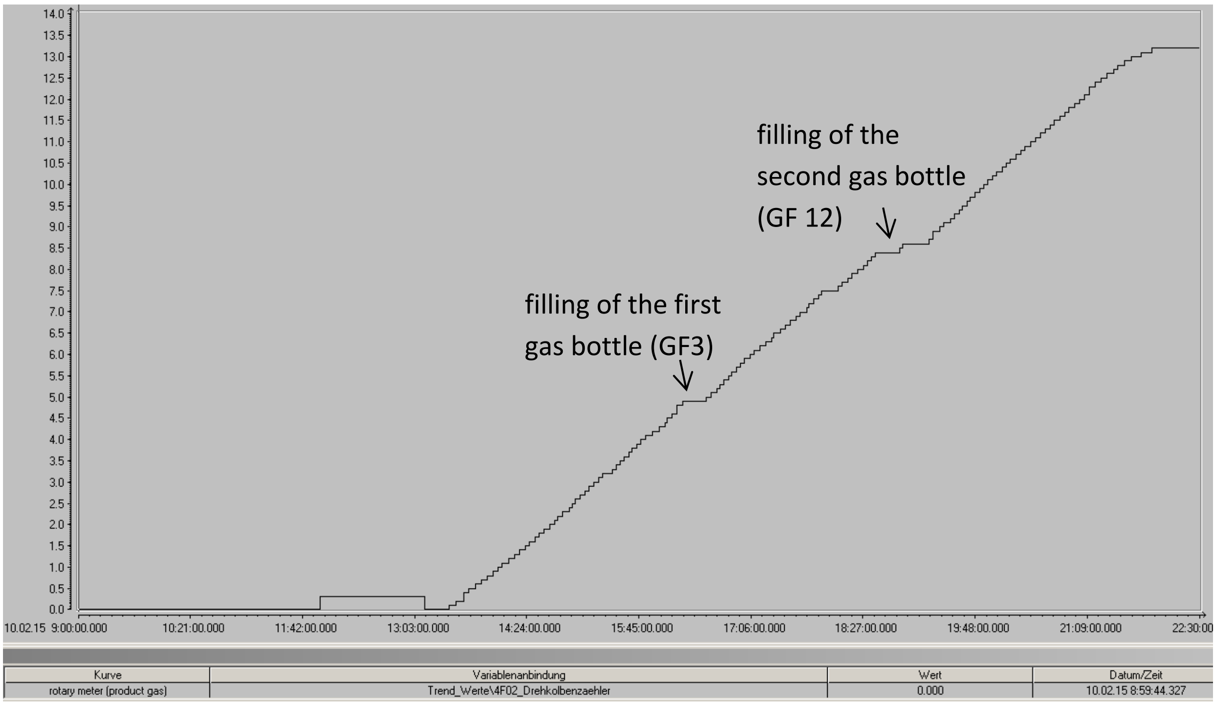


Figure 11: Amount of product gas in Nm^3/h.

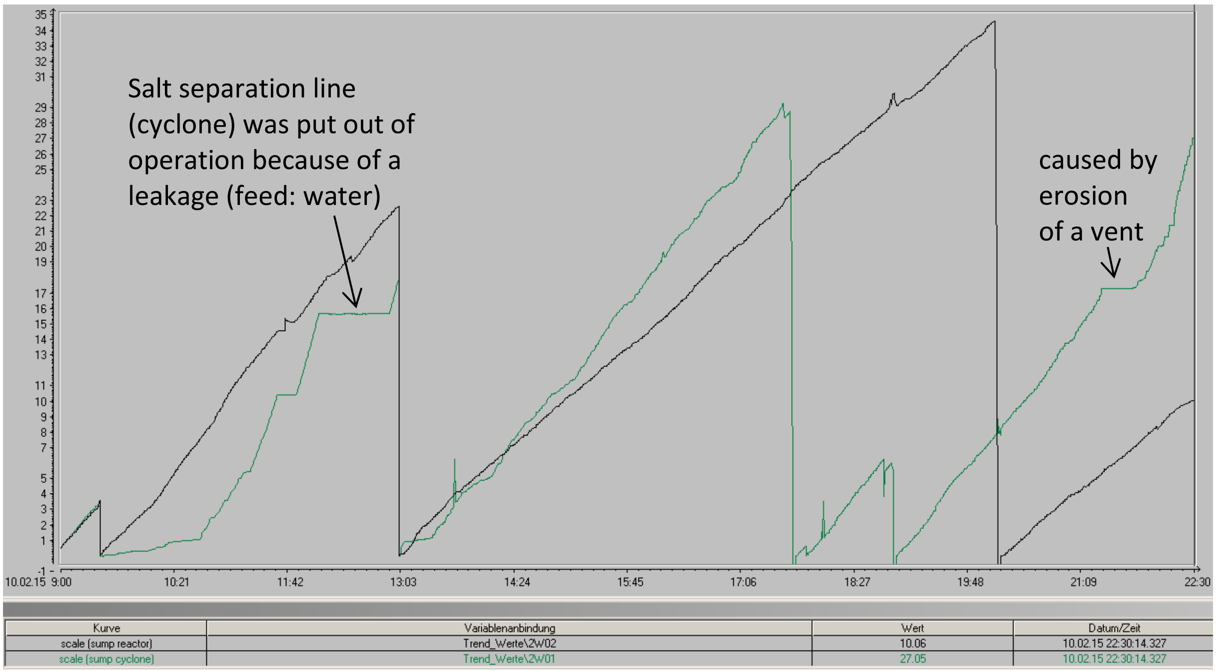


Figure 12: Sum of the salt concentrate separated from the sump of the cyclone (green) and from the sump of the reactor (black) in kg.

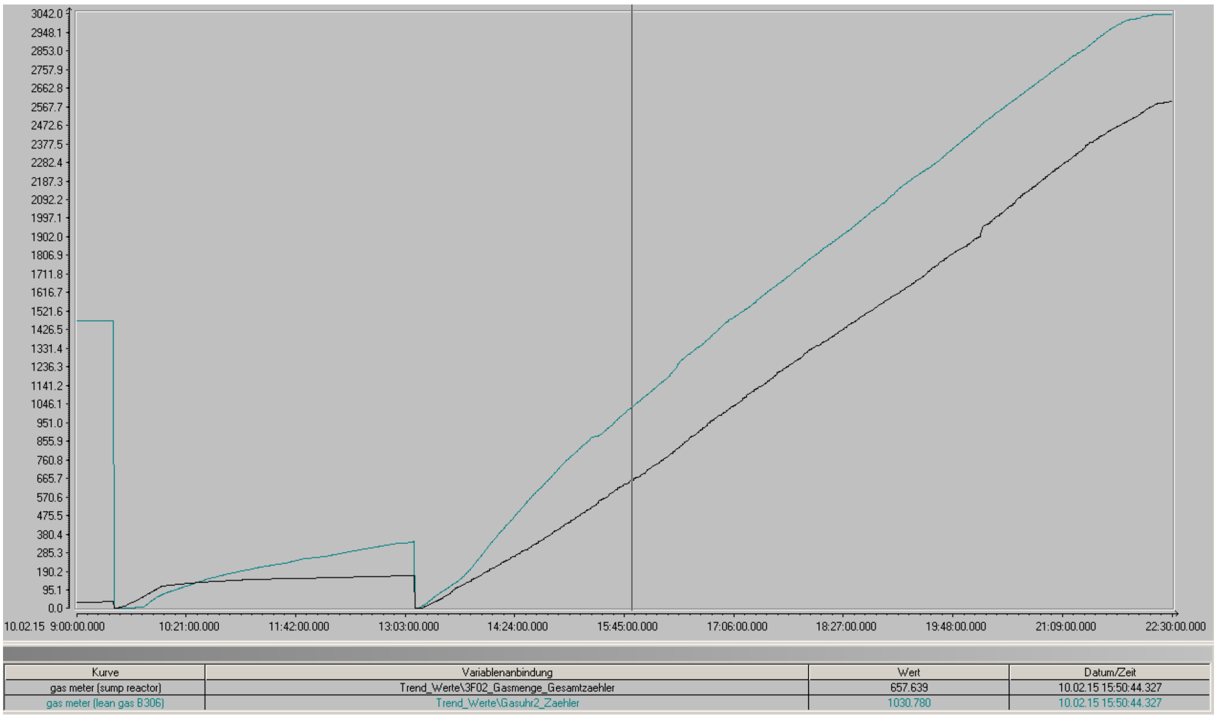


Figure 13: Sum of the lean gas from the waste water (turquois) and the salt concentrate separated from the sump of the reactor (black) in liter.

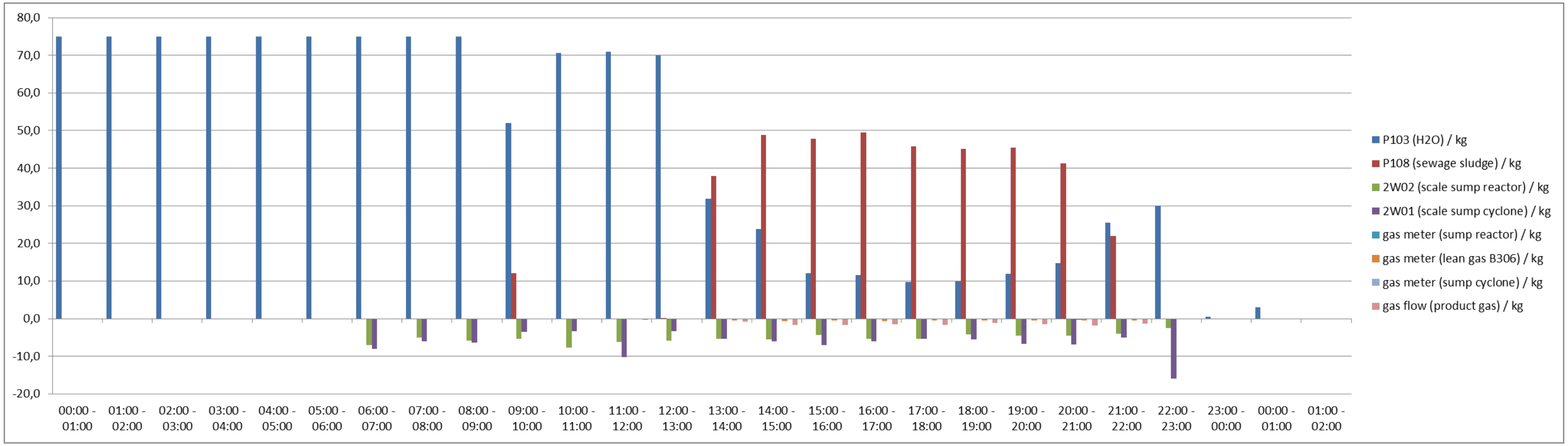


Figure 14: IN streams (positive values) and OUT streams (negative values) of the VERENA test KS-1 on Tuesday, the 10th February 2015 (without the effluent water).

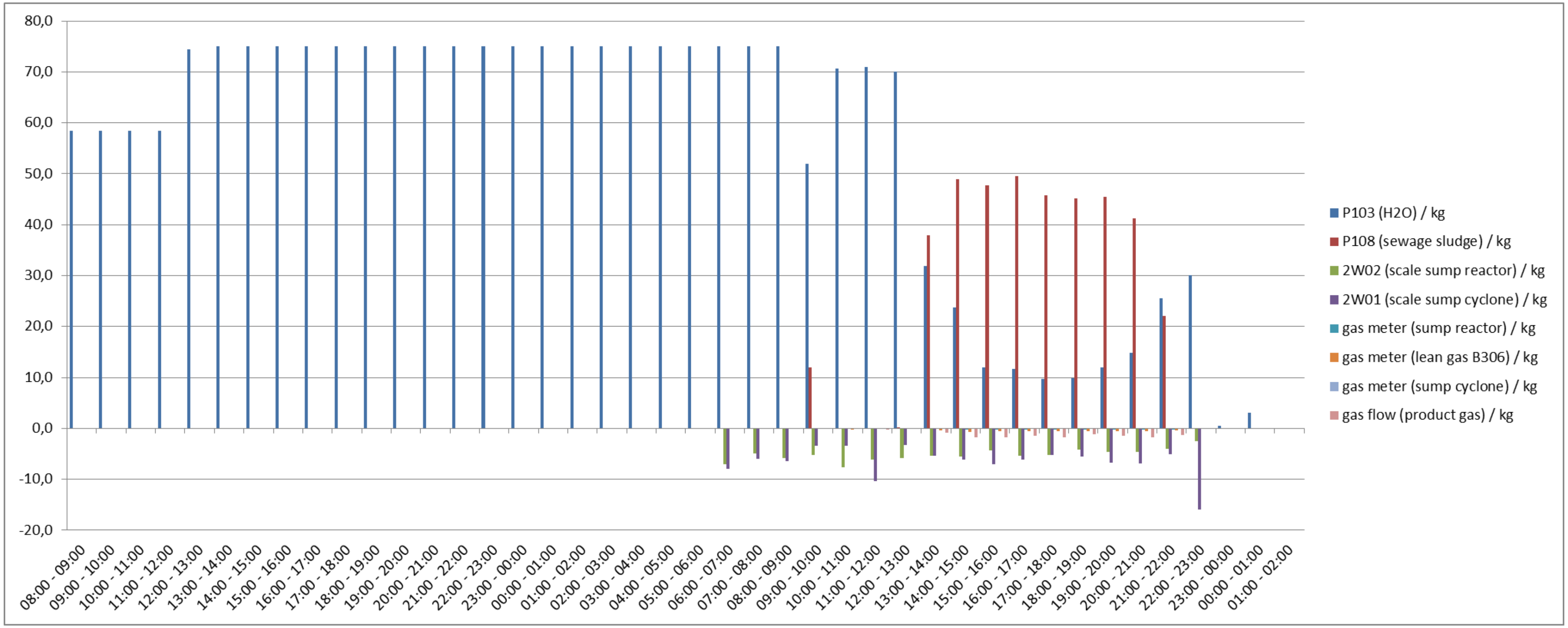


Figure 15: IN streams (positive values) and OUT streams (negative values) of the VERENA test KS-1 (without the effluent water) for the whole experimental time (including the heat up phase).

Table 22: EDX integral analysis of the residue of the plugging in the VERENA plant.

Element	Weight-%
C	66.00
O	10.61
Al	0.59
Si	0.78
P	0.78
S	0.81
K	3.07
Ca	0.73
Cr	4.89
Fe	3.33
Ni	5.62
Nb	0.52
Mo	2.26
Total	100.00

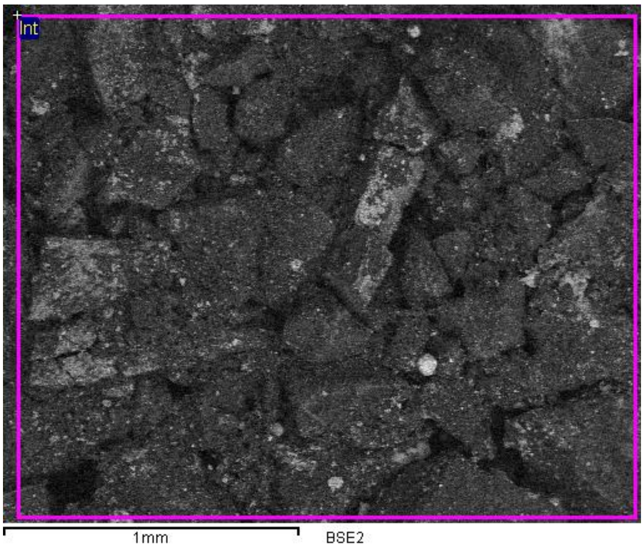


Figure 16: REM-picture of the residue of the plugging in the VERENA plant.

In Table 23 and Figure 17 the EDX/REM results of the analysis of some solid residue collected at the end of the reaction system are shown. It consists of corrosion products and salts and had a dark grey colour. The total mass was low and this should be understood only as indication of some corrosion products. This means there is some corrosion.

Table 23: EDX analysis of some solid residue in the VERENA plant.

Element	Weight-%
C	31.14
O	18.23
Al	0.35
Si	0.52
P	0.21
S	3.26
K	0.35
Ca	0.13
Ti	0.16
Cr	14.48
Fe	2.40
Ni	25.51
Nb	2.05
Mo	1.21
Total	100.00

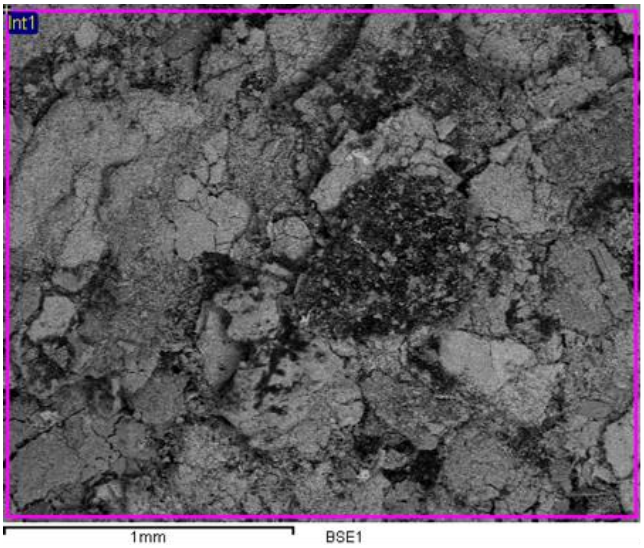


Figure 17: REM-picture of some solid residue in the VERENA plant (corrosion).

4.4.3 Experimental results

Table 24 shows the carbon balance under operating conditions. The balance period from 3:45 pm to 9:10 pm was selected according to constant flow rate of the feed (see Figure 10) and gas production rate $\dot{V}_{Gas}$ (see Figure 11). $Feed_{total}$ is the total pumped amount of sewage sludge within the duration of the balance period. C_{total} is the total amount of carbon in $Feed_{total}$. Thus the TOC and TIC amount of the feed as well as the carbon input via $KHCO_3$ is taken into account. The TOC-, NH_4^+-, TN_b-values of the effluent water are averaged values from three individual measurements. The carbon yield Y_C specifies the amount of carbon in the gaseous and liquid phases during constant operating conditions compared to the Ctotal in percent (accumulation areas within the plant are not taken into account).

Experiment conditions		
		Unit
Dry matter content	11,8	%
K+-conc. ($KHCO_3$)	2242	mg/l
mean reactor temperature	654	°C
pressure	280	bar
mean residence time	1,8	min
mean Feed mass flow	45,3	kg/h

Also for the two pilot scale experiments KS1 and KS2 the balance was calculated in two different ways. First is during steady state operation which gives the most accurate results for gasification yield TOC, NH4+ and TNb values in the waste water.

Second balance is performed for the total operation starting with first sewage sludge feeding up to the end of the experiment. This calculation gives also results on the salts, which are accumulated in the system and came out with delay.

Sewage sludge		
Time	5,42	h
Total sludge fed	245,4	kg
Total water fed additionally	63,4	kg
C- mass in total feed*	12,3	kg
TOC in Feed	5,0	%
Dry matter**	11,8 / 9,4	wt.-%
Total mass effluent	234,2	kg
Total mass salt concentrate liquid	22,5	kg
Total mass salt concentrate solid	11,4	kg
Total mass sump reactor	26,5	kg
Total mass total gas	13,0	kg
TOC-concentration		
in the effluent	2346	mg/kg
in the Salt conc. cyclone	14228	mg/kg
in the Salt conc. reactor	1545	mg/kg
Gas production – product gas	1,80	Nm^3/h
Gas production – total gas	2,39	Nm^3/h
Conversion of gasification	53,5	%
TOC-conversion***	89,9	%
Mass-balance	100	%
C-balance (TC)	70,4	%
N-balance	103,8	%
Product gas composition		
H_2	18,81	Vol %
CO	2,13	Vol %
CO_2	23,86	Vol %
CH_4	39,65	Vol %
C_2H_4	0,54	Vol %
C_2H_6	13,83	Vol %
C_3H_6	0,05	Vol %
C_3H_8	0,2	Vol %

*TIC in the feed not detectable

**original and after dilution in the HP feeding system

***without the C from $KHCO_3$

Table 24: Results of the steady-state phase

No.	Duration of the phase	Amount of Feed	C_{total}	Plugging	Y_{Gas}	Y_C	TOC-destruc-tion	Cold gas efficiency η	TOC-waste water	NH_4^+-waste water	TNb-waste water
[-]	[h]	[kg]	[kg]	[after h]	[%]	[%]	[%]	[-]	[mg/l]	[mg/l]	[mg/l]
KS1	5,42	245,4	12,3	9	53,5	70,4	89,9	0,61	2346	5750	3974

Y_{Gas} = conversion of gasification; Y_C = C-balance (TC)

Table 25: Results of analysis of experiment KS1 (steady state phase: 3:45 pm to 9:10 pm (10th February)

Aquon values do not fit this sheet and are listed in the Appendix. The solids were separated by centrifugation.

	Feed		aq. effluent (Position 7)				salt concentrate of cyclone (Position 2)					salt concentrate of reactor (Position 4)			
Parameter	Concentra-tion in dry matter	concentration in diluted sludge*	sample AA3 3:00 pm	sample AA4 5:00 pm	sample AA5 7:00 pm	total B306	sample AZ3 3:00 pm	sample AZ4 5:00 pm	sample AZ5 7:00 pm	total liquid B202	solid	sample AS3 3:00 pm	sample AS4 5:00 pm	sample AS5 7:00 pm	total B204
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]
TC	424	50032	4803	6182	5638	5054	13061	14931	13611	14425	137	5266	5025	4821	2544
TIC	n.d.	n.d.	2507	3222	3341	3205	n.d.	n.d.	n.d.	n.d.	n.d.	2853	3323	3329	907
TOC	424	50032	2295	2960	2297	1849	13061	14931	13611	14425	137	2413	1702	1492	1637
H	60,3	7115	-	-	-	-	-	-	-	-	17,6	-	-	-	-
N	37,3	4401	-	-	-	-	-	-	-	-	n.d.	-	-	-	-
O	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
NO3	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-	22,9	n.d.	n.d.	n.d.
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-	n.d.	n.d.	n.d.	n.d.
NH4	-	-	4530	5500	6100	5750	2000	2390	2640	1550	-	4700	5820	5700	1720
TNb	-	-	3467	4133	4016	4005	2704	3492	3573	2671	-	3716	4024	4073	1308
P	18,8	2218	4,30	n.d.	n.d.	2	254	262	350	145	84,1	61,8	47,5	20,7	43
PO4	-	-	38,1	n.d.	n.d.	n.d.	803	857	1052	725	-	153	135	73,4	117
S	6,11	721	37,5	64,4	70,8	84,8	1090	998	688	360	4,33	53,1	54,6	65,6	87
SO4	-	-	18,72	20,5	20,1	21,3	2014	2042	1340	936	-	n.d.	19,3	18,3	77,1
Ca	10,8	1274	0,42	0,13	0,04	0,19	7,4	4,75	3,4	6,45	49,2	0,20	0,17	0,14	2,25
K	20,5	2419	65,0	42,0	39,5	41,5	11365	11320	8040	3740	61,8	871	364	114	537
Mg	2,37	280	0,60	0,33	0,26	0,42	61,1	30,8	59,3	36,8	10,6	8,54	10,90	4,00	10,6
Na	0,972	115	4,10	3,60	4,40	4,4	105	105	76,9	43,2	4,02	38,8	5,60	61,8	13
Si	22,9	2702	35,0	29,5	37,3	32,6	107	118	127	94	89,5	29,4	29,7	31,0	25,5
Al	10,3	1215	0,40	0,41	0,39	0,44	n.d.	n.d.	n.d.	n.d.	44,8	0,63	0,14	0,10	0,15
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

	Feed		aq. effluent (Position 7)				salt concentrate of cyclone (Position 2)					salt concentrate of reactor (Position 4)			
Parameter	Concentra-tion in dry matter	concentration in diluted sludge*	sample AA3 3:00 pm	sample AA4 5:00 pm	sample AA5 7:00 pm	total B306	sample AZ3 3:00 pm	sample AZ4 5:00 pm	sample AZ5 7:00 pm	total liquid B202	solid	sample AS3 3:00 pm	sample AS4 5:00 pm	sample AS5 7:00 pm	total B204
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]
Cu	0,317	37,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1,49	n.d.	n.d.	n.d.	n.d.
Pb	0,054	6,372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,22	n.d.	n.d.	n.d.	n.d.
Zn	0,683	80,6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	3,08	0,18	n.d.	n.d.	n.d.
Cr	0,037	4,366	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,42	n.d.	n.d.	n.d.	n.d.
Fe	16,5	1947	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	74,9	0,13	0,41	0,66	0,08
Mo	0,009	1,062	n.d.	n.d.	n.d.	n.d.	12,8	5,75	2,84	8,4	0,333	24,4	2,5	0,51	2,2
Ni	0,020	2,36	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1,39	5,6	0,48	n.d.	n.d.
Cl	5,17	610	86,3	83,8	78,4	81,2	4598	4875	3148	2009	0,423	124	132	87,1	166
Hg	0,00025	0,0295	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,0003	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	288	345	294	265	178	215	217	169	-	302	316	308	106

n.d. Limits are listed in the Appendix.

Legend:	n.d.	not detected

		Feed	cyclone solid	
Ash content	by 550°C:	20,17	81,54	%
	by 815°C:	19,94	80,52	%
	by 1000°C:	19,15	79,61	%
Higher Heating value:		18308	-	J/g

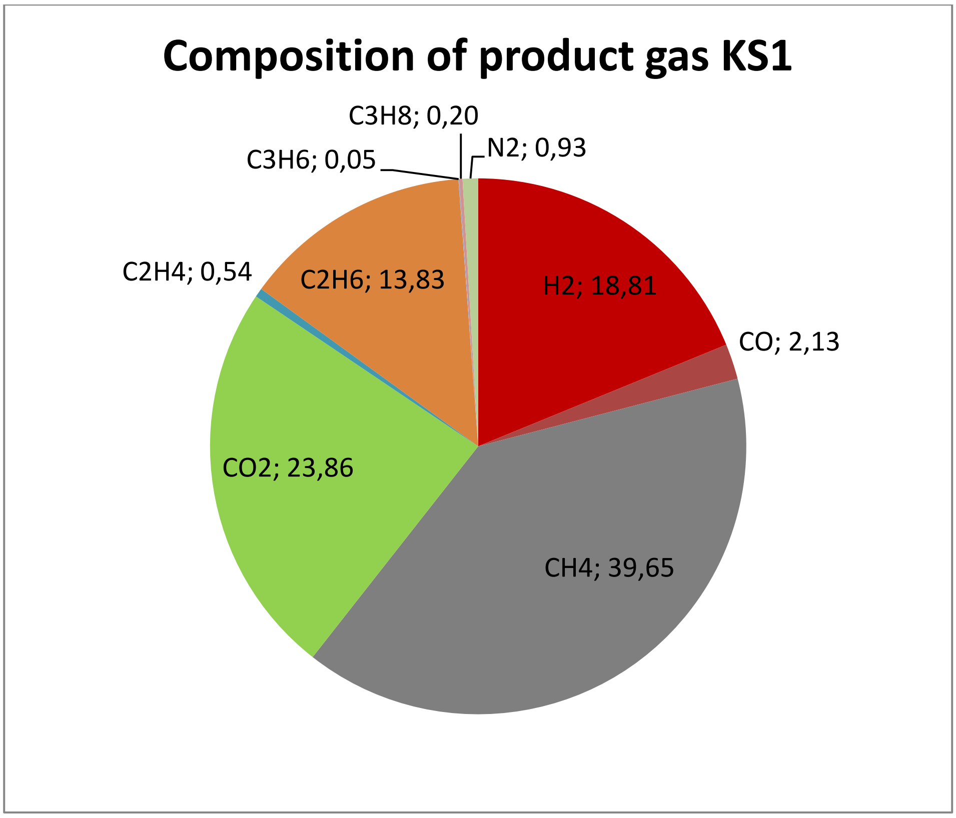


Figure 18: Composition of the product gas of experiment KS1.

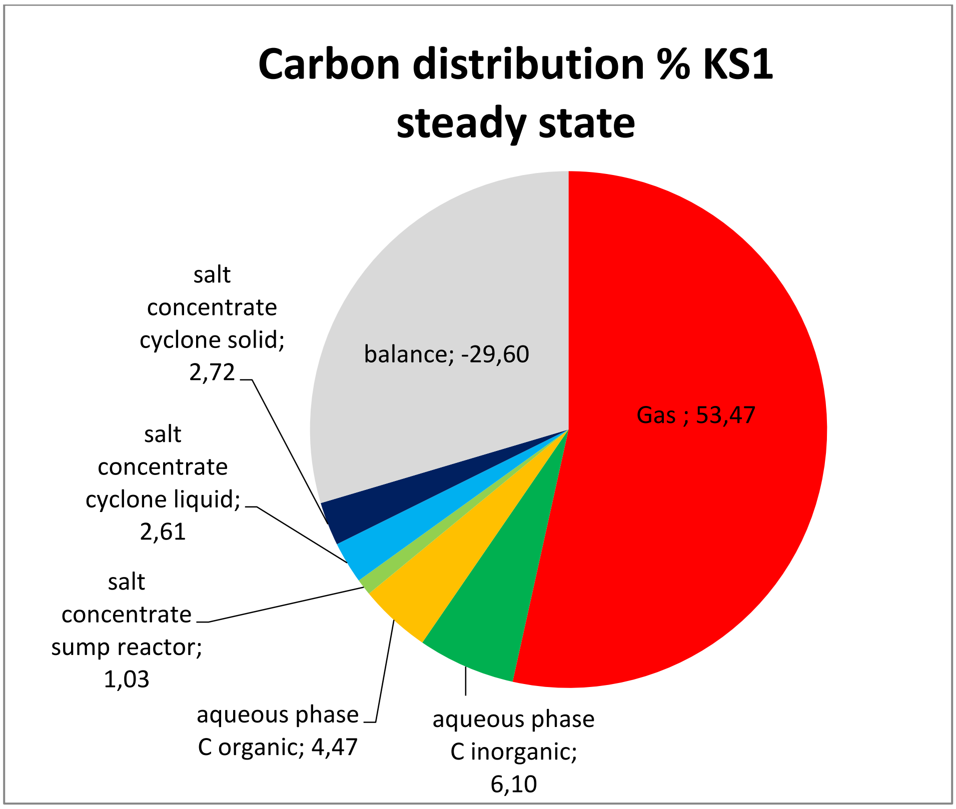


Figure 19: Carbon distribution of experiment KS1 during the steady state phase.

Table 26: composition of the lean gas and product gas of experiment KS1.

date	time	x_{H2} [vol%]	x_{CO} [vol%]	x_{CH4} [vol%]	x_{CO2} [vol%]	x_{C2H4} [vol%]	x_{C2H6} [vol%]	x_{C3H6} [vol%]	x_{C3H8} vol%]	x_{N2} vol%]	sum	Kommentar
lean gas sump cyclone B201												
10.02.2015	15:00	14,00	8,68	2,39	72,09	0,96	0,71	0,92	0,25	-	100,00	nitrogen was not measured
10.02.2015	17:00	11,76	8,06	4,54	70,98	1,27	1,34	1,33	0,72	-	100,00	
10.02.2015	19:00	12,64	7,82	7,18	65,45	1,85	2,19	1,88	1,00	-	100,00	
10.02.2015	21:00	13,91	6,97	8,02	63,99	1,86	2,36	1,89	0,99	-	100,00	
10.02.2015	23:15	26,95	5,72	8,46	53,39	1,49	1,87	1,42	0,70	-	100,00	
lean gas effluent B306												
10.02.2015	16:00	10,52	0,51	11,78	74,53	0,31	2,27	0,05	0,04	-	100,00	nitrogen was not measured
10.02.2015	18:00	10,10	0,30	11,16	76,02	0,22	2,15	0,02	0,02	-	100,00	
10.02.2015	20:00	10,57	0,20	10,89	76,05	0,22	2,04	0,02	0,01	-	100,00	
10.02.2015	22:00	14,39	0,27	11,61	71,09	0,26	2,34	0,02	0,02	-	100,00	
10.02.2015	23:15	13,91	0,31	11,71	71,38	0,25	2,42	0,02	0,01	-	100,00	
lean gas sump reactor B203												
10.02.2015	15:00	17,63	0,36	32,90	35,64	0,42	11,79	0,03	0,45	0,77	100,00	
10.02.2015	17:00	16,74	0,96	34,54	36,03	0,24	11,42	0,00	0,07	0,00	100,00	
10.02.2015	19:00	17,14	0,82	34,46	36,08	0,17	11,32	0,00	0,02	0,00	100,00	
10.02.2015	21:00	18,20	0,88	35,86	32,39	0,16	10,50	0,00	0,02	2,00	100,00	
10.02.2015	23:15	30,27	0,57	26,56	33,21	0,12	8,25	0,00	0,00	1,02	100,00	
product gas B301												
10.02.2015	18:00	18,81	2,13	39,65	23,86	0,54	13,83	0,05	0,20	0,93	100,00	
10.02.2015	20:00	19,50	1,83	39,53	25,02	0,45	13,55	0,00	0,11	0,00	100,00	
10.02.2015	22:00	22,10	1,43	38,70	24,10	0,41	13,17	0,00	0,09	0,00	100,00	
11.02.2015	09:00	11,83	0,57	18,75	10,71	0,20	6,34	0,00	0,00	51,59	100,00	gas that remained in B301 (after experiment)
product gas of the gas bottles												
10.02.2015	16:00	19,53	1,68	40,63	23,25	0,64	13,84	0,15	0,29	-	100,00	
10.02.2015	19:30	20,58	0,98	40,21	24,43	0,47	13,10	0,07	0,16	-	100,00	
11.02.2015	09:00	15,72	1,10	25,41	14,44	0,28	4,07	0,00	0,00	38,98	100,00	gas that remained in B301 (filling of GF7 after experiment)

Table 27: Lean gas of the salt concentrate separated from the sump of the cyclone (offline data)

date	time	cyclone gas meter / NL
10.02.2015	08:10	-
10.02.2015	09:00	6
10.02.2015	13:05	22
10.02.2015	14:05	17
10.02.2015	15:00	38
10.02.2015	16:05	64
10.02.2015	17:00	53
10.02.2015	18:00	71
10.02.2015	19:00	73
10.02.2015	20:00	74
10.02.2015	21:00	72
10.02.2015	22:00	50
10.02.2015	23:15	44

Besides the balance during the steady state operation - which reflects the operation of a commercial plant working all around the clock - a second kind of balance called here as total balance (as mentioned before) can be performed. Some additional information about the distribution of the inorganic salts can be gained. During lab-scale operation, a better balance has also been achieved. This is only the case if all aqueous solution used to clean the plant are measured and analyzed. Since this is not practical for the large pilot plant, the informative value of the “total balance” for the two pilot plant experiments is low. Nevertheless, the total balance is presented here to complete the picture.

Table 28: total masses of all in and out streams of the VERENA plant during experiment KS1 (from the beginning of the first feeding of sewage sludge till the end of the experiment).

evaluation period for total balance		09:00 am -11:00 pm 10.02.15	Only for B306 balance 1:10 pm to 11:00 pm	
Feed **DM Feed**		11,80		wt.-%
Feed (wet) **total**		402000	402000	g
H2O total		836000		g
effluent and salt concentrate				
total effluent (calculated!)		1049275,41	474800	g
salt concentrate:				
liquid sump reactor		72800		g
liquid sump cyclone		69300		g
solid sump cyclone	wet	22500		g
total sump cyclone		91800,00		g
total gas		24335,59		g

Table 29: total amount of all gas streams of the VERENA plant during experiment KS1 (from the beginning of the first feeding of sewage sludge till the end of the experiment).

Gasphase	**C-amount**		**gas amount:**	
B306 (measured by a gas meter)	1604,8	g C	5248,98	g
B203 (measured by a gas meter)	963,3	g C	2154,05	g
B201 (measured by a gas meter)	296,8	g C	940,56	g
product gas (rotary meter and gas bottles)	7926,6	g C	15992	g
total:	**10791,5**	g C	**24335,59**	g

Notice: The vessel B306 does not represent a cumulative sample of the whole process like the other vessels, but is a throughput vessel with a long residence time of about 1 to 2 hours. So balance of the aqueous phase is performed not for the whole time period, but only from 13:10 to 23:00.

Table 30: element balance of non-metal elements.

Element	N		P		S		Cl		Si	
	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %
Feed	1769,36		891,80		289,83		245,24		1086,28	
aqueous phase	1901,57	107,47	0,95	0,11	40,26	13,89	38,55	15,72	15,48	1,42
salt concentrate sump reactor	95,22	5,38	3,13	0,35	6,33	2,19	12,10	4,93	1,86	0,17
salt concentrate cyclone liquid	185,10	10,46	10,01	1,12	24,95	8,61	139,19	56,76	6,51	0,60
salt concentrate cyclone solid	0,00	0,00	404,26	45,33	20,81	7,18	2,03	0,83	430,22	39,60
total	2181,90	123,32	418,35	46,91	92,36	31,87	191,87	78,24	454,07	41,80

Table 31: element balance of metal-elements.

Element	K		Ca		Mg		Fe		Al	
	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %
Feed	972,44		512,31		112,42		782,69		488,59	
aqueous phase	19,70	2,03	0,09	0,02	0,20	0,18	0,00	0,00	0,21	0,04
salt concentrate sump reactor	39,09	4,02	0,16	0,03	0,77	0,69	0,01	0,00	0,01	0,00
salt concentrate cyclone liquid	259,18	26,65	0,45	0,09	2,55	2,27	0,01	0,00	0,00	0,00
salt concentrate cyclone solid	297,07	30,55	236,50	46,16	50,95	45,32	360,04	46,00	215,35	44,08
total	615,05	63,25	237,20	46,30	54,47	48,45	360,06	46,00	215,57	44,12

Table 32: C-balance of KS1 (total balance)

Element	C	
	Mass, g	Recovery, %
Feed	20112,86	
Gas	10791,50	53,65
aqueous phase C organic	877,91	4,36
aqueous phase C inorganic	1521,73	7,57
salt concentrate sump reactor	185,20	0,92
salt concentrate cyclone liquid	999,65	4,97
salt concentrate cyclone solid	658,55	3,27
total	15034,54	74,75

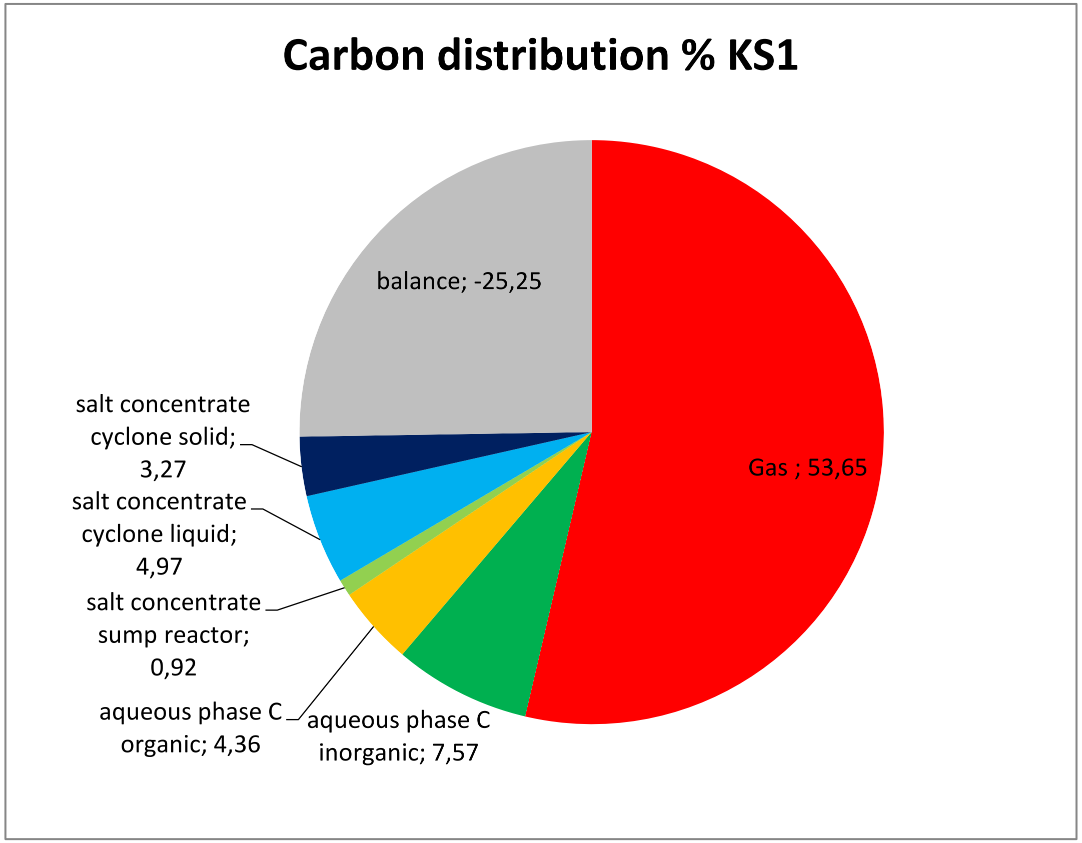
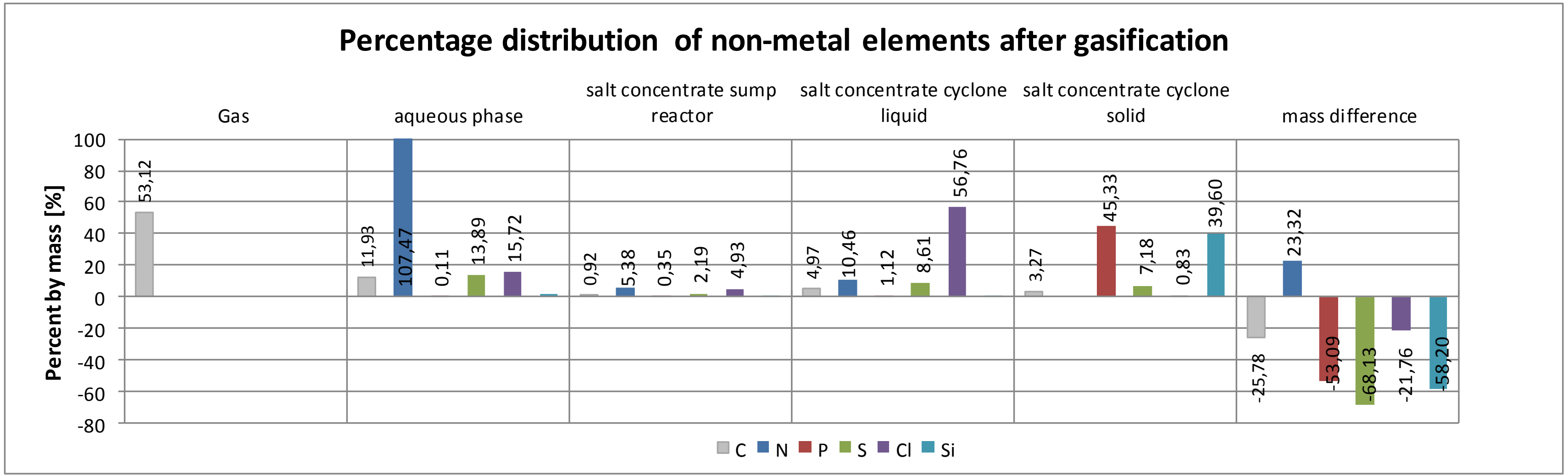
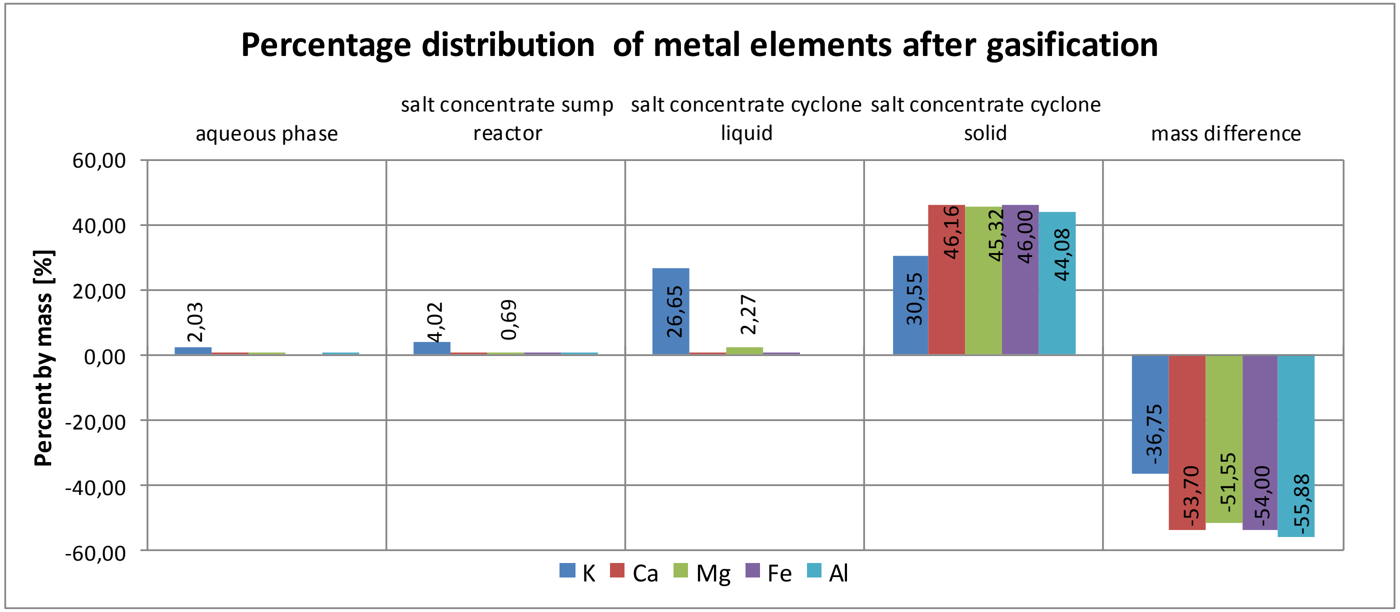


Table 33: Comparison of conversion of gasification and TOC-conversion of the total balance and during steady state operation.

	conversion of gasification [%]	TOC-conversion [%]
total balance	53,7	86,8
steady state operation	53,5	89,9


Percentage distribution of non-metal elements after gasification
Gas
aqueous phase
salt concentrate sump reactor
salt concentrate cyclone liquid
salt concentrate cyclone solid
mass difference
Percent by mass [%]
100
80
60
40
20
0
-20
-40
-60
-80
53,12
11,93
107,47
0,11
13,89
15,72
0,92
5,38
0,35
2,19
4,93
4,97
10,46
1,12
8,61
56,76
3,27
45,33
7,18
0,83
39,60
-25,78
23,32
-53,09
-68,13
-21,76
-58,20
C
N
P
S
Cl
Si


Percentage distribution of metal elements after gasification
aqueous phase
salt concentrate sump reactor
salt concentrate cyclone liquid
salt concentrate cyclone solid
mass difference
Percent by mass [%]
60,00
40,00
20,00
0,00
-20,00
-40,00
-60,00
2,03
4,02
0,69
26,65
2,27
30,55
46,16
45,32
46,00
44,08
-36,75
-53,70
-51,55
-54,00
-55,88
K
Ca
Mg
Fe
Al

In Table 34 the balance of minor elements is shown. The limit of quantification of liquids is dependent from the dilution factor. Since the liquid samples often contain a lot of salts and organics the samples also have to be diluted for minor analysis. Therefor the limits of quantification (see Appendix) are accordingly high.

The increased Mo, Ni and Cr-values indicate corrosion.

Table 34: balance of minor elements.

Element	Na		Cu		Pb		Zn		Cr		Mo		Ni		Hg	
	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %
Feed	46,11		15,04		2,56		32,40		1,76		0,43		0,95		0,012	
aqueous phase	2,09	4,53	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,000	0,000
salt concentrate sump reactor	0,95	2,05	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,16	37,52	0,00	0,00	0,000	0,000
salt concentrate cyclone liquid	2,99	6,49	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,58	136,35	0,00	0,00	0,000	0,000
salt concentrate cyclone solid	19,32	41,91	7,16	47,63	1,04	40,72	14,81	45,70	2,03	115,85	1,60	374,94	6,68	704,27	0,001	12,160
total	25,35	54,98	7,16	47,63	1,04	40,72	14,81	45,70	2,03	115,85	2,34	548,81	6,68	704,27	0,001	12,160

In Table 35 the ash content of the feed and the salt concentrate separated from the sump of the cyclone are listed. 43 % of the ash content of the feed was separated by the cyclone. No ash separation from the reactor and the waste water was performed.

Table 35: Ash content of the Feed and the salt concentrate separated from the sump of the cyclone.

Ash in total Feed	9083,99	g
Ash in salt concentrate cyclone	3904,87	g
Ash salt concentrate/Ash Feed	42,99	%

.

Additional analysis of the product gas

Table 36: results of the tar measurement of the product gas.

20 ml Wasser/Ethylenglykol 9:1 6 °C, 30 min, ~ 1,5 Nl	Blind, 06.02.15		Blind, 06.02.15		VERENA 10.02.15, 19:00		VERENA 10.02.15, 20:00	
	S3		S4		S5		S6	
	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3
Benzene	**0,29**	0,20	**0,81**	0,54	**2307**	1538	**4668**	3112
Toluene	**7,97**	5,31	**3,88**	2,58	**1116**	744	**1808**	1205
Naphthalene	**0,05**	0,04	**0,08**	0,06	**1,4**	0,9	**3,5**	2,3

The product gas samples also contain xylenes, ethyl benzene, styrene and indene.
A reason for the higher values of the second measurement could be a possible absorption at the tubes during the first measurement (after saturation we measure the higher values).

The H_2S and NH_3 measurement of the product gas showed the following concentrations: 0,1 vol% H_2S and <0,0001 vol% NH_3.

In Table 37 and Table 38 the Hg-measurements done by Tauw B.V. according to NEN-EN 13211: 2001 Norm are listed. The amount of Hg (0,006 mg/Nm^3) in the process gas is very low.

Table 37: results of the Hg measurement of the process gas done by Tauw B.V.

Results measurements Mercury - Karlruher Institüt für Technologie - process

Parameter	unit	measurement 1	measurement 2	measurement 3	average
date	[dd-mm-yyyy]	11-02-2015	11-02-2015	11-02-2015	
time start	[hh:mm]	14:20	14:56	15:32	
time stop	[hh:mm]	14:50	15:26	16:02	
mercury	[mg/Nm³]	0,004	0,008	0,006	0,006

Table 38: results of the Hg measurement of the product gas from gas bottle GF12 done by Tauw B.V.

Results measurements Mercury - Karlruher Institüt für Technologie - gas bottle

gegevens	eenheid	measurement 1	measurement 2	measurement 3	average
date	[dd-mm-yyyy]	11-02-2015	11-02-2015	11-02-2015	
time start	[hh:mm]	14:23	14:56	15:32	
time stop	[hh:mm]	14:53	15:26	16:02	
mercury	[mg/Nm³]	0,002	0,002	0,001	0,002

6 Pilot scale experiment KS2

The second pilot plant experiment was started for two times because during start-up operation with water the plant showed again clogging. The plug was removed but after discussion with the Dutch delegation present, the feeding of sewage sludge did not start because of the uncertainties concerning the solids in the reaction system.

For the second start of experiment KS2 the concentration of the sewage sludge was reduced in order to avoid some of the problems with KS1 (viscosity, high salt load) and enchance the gasification rate. In the following the run and the results of the pilot-scale test in the VERENA facility are presented.

6.1 Pretreatment

The sewage sludge from Oijen was delivered in 15 plastic vessels which were filled with dewatered sludge cake and centrate liquid from the dewatering process. The content of the barrels is shown in Table 39.

Table 39: Amount of dewatered sludge cake and centrate liquid in the delivered vessels.

nr.	dewatered sludge cake (kg)	Centrate liquid (kg)	Dry matter % in vessel
1	40,0	110,0	6,9
2	40,0	110,0	6,9
3	39,8	110,0	6,9
4	40,0	110,0	6,9
5	39,8	110,0	6,9
6	40,0	110,0	6,9
7	40,0	110,0	6,9
8	40,4	110,0	7,0
9	40,2	110,0	7,0
10	40,0	110,0	6,9
11	40,0	110,0	6,9
12	40,0	110,0	6,9
13	40,6	110,0	7,0
14	40,0	110,0	6,9
15	40,0	110,0	6,9

The content of every vessel was first stirred (see Figure 20a) and then pumped into the tank of a screw pump. Parallel to each barrel 5,5 L $KHCO_3$ solution were added, which results in a concentration of 2500 mg K^+/L since potassium salts catalyze the gasification reaction (K^+ is added at a concentration of 2500 mg/L sludge additionally to the starting K value of the sludge). The sludge with the $KHCO_3$ solution was recirculated for homogenization and subsequently conveyed into an IBC (see Figure 20b).

Figure 20: Pretreatment of the feed.

In Table 40 it is listed in which of the 3 IBCs which vessel was filled.

Table 40: Assignment of the barrels to the IBCs.

IBC No.	vessel No.	DM % in IBC (30.04.15)	DM % in IBC (04.05.15)
1	2, 5, 6, 8, 10	8,57	
2	3, 4, 7, 9, 11	7,23	7,49
3	1, 12, 13, 14, 15	7,12	

To determine the dry matter content of the 3 sewage sludge charges respectively ca. 200 g sewage sludge were dried in an oven at 105 °C for 24 h. A double determination of the dry matter of every charge was executed.

It was noticed that the dry matter content 8,57 wt.-% of IBC No. 1 was high compared to the values of IBC No. 2 and IBC No. 3, since the sludge of the single barrels almost had the same dry matter content. Also the values of the double determination exhibited a too high difference. Therefor it was planned to take a new feed sample of the IBC No. 1 directly before feeding into the plant. However this determination was not carried out because the sludge of IBC No. 1 was not pumped into the pilot plant due to a premature end of the experiment. Treatment with the colloid mill was not performed, due to the very fine consistence of this sludge, much finer than the 1 mm which can be set to the colloid mill. Also the homogenization of the feed (each barrel delivered had two phases; a paste like sludge and water phase) was performed by stirring. During KS1 this was not possible due to the high viscosity of the mixture.

6.2 Parameters of the experiment

The pilot plant VERENA was operated at a pressure of 270 bar (only during the filling of the high pressure bottles the pressure declined temporary a little bit – lowest value 222 bar), while the lab-scale apparatus LENA was operated at 280 bar during all 15 experiments with sewage sludge. The operation pressure of 270 bar instead of 280 bar was chosen to reduce

the risk of a rupture disk bursting. However it has already been shown, that a pressure variation in this range has no significant influence on the reaction. In Table 41 the parameters of the experiment are summarized. The selection of the experimental parameters is based on the results of the lab scale tests and the first pilot scale test (KS1). Instead of sludge with 11,8 wt.-% for experiment KS1, sludge with 7,49 wt.-% was fed into the plant -as mentioned- because during the first pilot-scale experiment the high viscosity of the concentrated sludge caused frequent to interruptions of the process. Also during experiment KS1 the targeted feeding rate of 75 kg/h was not achievable, most probably due to visible gas bubbles in the feed slurry. These are also the reasons why the desired feeding rate for the second pilot-scale experiment was lowered to 50 kg/h.
The trends of the temperatures in the reactor are depicted in the Appendix.

Table 41: Experimental parameters of the pilot-scale test

No.	Type	Dry Matter of the Feed-sludge	pressure	$T_{Preheater}$ [1]	$T_{Reactor}$ [2]	Reactor- mean residence time	Feed (sludge)
				(outlet)	(midpoint)		
[-]	[-]	[wt-%]	[bar]	[°C]	[°C]	[min]	[kg/h]
KS2	Oijen	7,49	270	477	640	2,6	50,0

[1] $T_{Preheater}$ is the outlet temperature of the preheater of the salt separation system.
[2] $T_{Reactor}$ is the mean midpoint of the triple thermocouple within the reactor.

6.3 Detailed description and results of the experiment KS2

The experiment KS2 was evaluated in the same way as experiment KS1 (see chapter 4.4). Also the parameters were analysed analog to the first pilot-scale experiment.

6.3.1 Progress of experiment KS2

<u>3rd May</u>
The campaign already started on Sunday the 3rd May 2015 at 10:00 pm. During the heating phase the plant was operated at 250 bar and fed with 50 kg water per hour. The temperature of the two heating systems (electric heater W206 and propane gas burner A201, see Figure 8) was increased by about 60 °C/h.

<u>4th May</u>
At 5:10 am on Monday the lines for the separation of the salts from the sump of the cyclone and from the sump of the reactor were put into operation. 1 ¾ hours later the operation pressure was increased to 275 bar. To prepare the operation with sewage sludge the IBC No. 3 was brought into the VERENA hall at 7:38 am and the sludge was pumped in a circle for homogenization. At 2:15 pm the plant was in operation with water at high

temperatures for several hours and the reactor temperature was constant for nearly two hours. At this point of time the blind samples were taken.

Subsequently at 2:17 pm sewage sludge was pumped into the reaction system while the flow of water was first slowly reduced and afterwards stopped. However after about one hour later the line for the separation of salts from the sump of the cyclone was clogged. To prevent that the salts have to pass the preheater, which could cause plugging there, the sewage sludge pump was switched off. Again 50 kg/h water was pumped into the plant. After a short period of time there was suddenly a loud bump and the corresponding line was freed from clogging. Thereafter it was observed that the salt separation system runs faultlessly again.

For this reason the sewage sludge pump was switched on at 5:15 pm but didn't deliver the sludge. The pump P108 was flushed with water but this procedure did not solve the problem. The pump was even not able to convey water. Therefore the vent of the suction side of the pump was removed. The o-ring had a little defect and a small lump of hairs, like felt was found there. The vent seat showed little cracks. After the reparation of the vent of the suction side the pump still didn't work. So the pressure side of the pump was also removed and replaced. At 9:35 pm the pump P108 was repaired and delivered about 18 kg/h water into the reaction system. The pump P103 also conveyed water to result in a total water flow rate of ca. 50 kg/h. After the pump was operating constant for 1 ½ h, it was switched off and again prepared for conveying sewage sludge by connecting the corresponding hoses.

Shortly after the feeding of sewage sludge was restarted it was observed that the line for the separation of the salts from the sump of the cyclone was clogged (obviously from salt remained in the system). For this reason the pumping of sewage sludge was stopped again. It was tried to overcome the problem of clogging by switching to the parallel line for salt separation and bypassing the buffer tank B205. However this was not successful. Then this line was taken out of operation and flushed with nitrogen. Due to this procedure the line was released from plugging and the separation of salts from the sump of the cyclone was again taken into operation (the normal way over the buffer tank B205).

5th May

At 0:37 am on Tuesday the pump P108 was switched on and delivered sewage sludge into the pilot plant. But the flow rate of the sewage sludge was fluctuating strongly. The operating pressure of the plant was reduced from 275 bar to 270 bar. At 1:34 am the sewage sludge pump P108 was not conveying the feed anymore. The flow rate of water was increased to 50 kg/h (pump P103). It was observed that the IBC No. 3, was full of foam. So possibly the foam caused the problems of the pump P108. In order to reduce the foam, the hose for recirculation, which was attached to the lid of the IBC No. 3, was put directly into the sludge. Further attempts to receive a steady feed flow by the pump P108 failed. At 2:45 am the pump was switched off because it failed again. The vent of the pressure side was replaced for a second time. From 3:48 am to 4:42 am about 40 kg/h sewage sludge with pump P108 and 10 kg/h water with pump P103 were delivered into the plant. The feeding

was stopped again by the failure of pump P108. But after about 15 minutes later the pump could be restarted and again the plant was fed with ca. 40 kg/h sewage sludge and 10 kg/h water till about 6:10 am.
Due to the permanent failure of the pump P108 it was decided to deliver the sewage sludge with pump P103 into the plant. From that point of time on the pump P108 was used for the supply of pure water. The feeding with pump P103 started at 8:44 am. After about 10 minutes a flow rate of 50 kg/h sewage sludge was reached and the pump P108 was switched off.
At 9:26 am the pump P103 also had to be switched off because the level of the water phase inside the HP autoclave B301, where a value of about 40 % is usually kept, reached very fast a value of ca. 85 %. At the same time the gaseous phase has expanded normally in order to keep the pressure of the system constant. The origin of this dysfunction was tar formation in the low temperature part of the system autoclave and tubing. Due to the fact that both pumps were turned off, the temperature of the burner and the electric heater was lowered. After cleaning of the autoclave B301 and its tubing, the operation was started again at 10:51 am with water. The temperatures of the electric heater and the burner were increased to reach the desired temperatures again.
Accordingly at 3:45 pm sewage sludge was pumped into the reaction system (P103) while the flow of water was slowly reduced (P108). At 4:21 pm a flow rate of 50 kg/h sewage sludge was reached and the pump P108 was switched off. From 7:09 pm to 9:07 pm a first gas bottle was filled, while the filling of a second one started at 11:22 pm. Steady state operation was reached for 7,67 h. During this phase the corresponding samples were taken. At about 6:50 pm the throughput of the sump of the reactor decreased slowly. At 8:05 pm this line was clogged. Plant operation continued normal except the clogged sump of the reactor. During the experiment several attempts were made to release this system from clogging. The corresponding line was even two times taken out of operation, partly disassembled and flushed with compressed air. Later, the analysis showed that the solids separated there consisted mainly of corrosion products. Their concentration was too high for the system. This part of the system was overloaded (very dense solid material in the tubing). Furthermore the vent seat and the spindle of one of the pneumatic valves had to be exchanged.
After several hours of stable operation, the pressure in the entrance of the plant became higher (less than 10 bar) than the pressure in the exit part of the plant. This was an indication for general fouling that is not localized in one apparatus.

6th May

For this reason at 0:07 pm on Wednesday the flow rate of sewage sludge was reduced to 25 kg/h and 25 kg/h water were added to the feed stream in order to reduce the pressure differences. But this procedure didn’t help. The pressure differences rose steadily, that’s why at 1:03 am the feeding of sewage sludge was stopped and the plant was flushed with 50 kg/h water at operation temperature and pressure. This did not result in a reduced pressure difference and thus, the temperature was reduced (but still over 500 °C). From 1:44 pm to

3:14 pm the Tauw B.V. performed three Hg-measurements of the product gas collected in the second gas bottle (GF7). Direct measurement of the process gas was not possible because during this period of time the plant was run with water. After several hours (18 h) of operation with water, the pressure difference decreased and the plant was heated up again to reach again the reaction temperature of 640 °C.
As the temperature in the reaction system reached 630 °C, pressure loss was noticed. It was not possible to detect the position of the leak (no visible steam coming out of the isolation). After some minutes, the pressure loss escalated and the experiment was interrupted. Examination of the reaction system after experiment did not manage to localize the leak point exactly. This will be performed after the next experiments in the VERENA plant and disassembling of this part of the plant (for the next experiments the high pressure part of the main preheater is out of line). There is evidence that the leak is in the pre-heater (the HP coil becomes visible after disassembling a part of the heating system).

During the pilot-scale experiments delegation of STOWA visited the VERENA plant at KIT.

6.3.2 Experimental data KS2

In the following operation diagrams (screen shots) of experiment KS2 are shown.

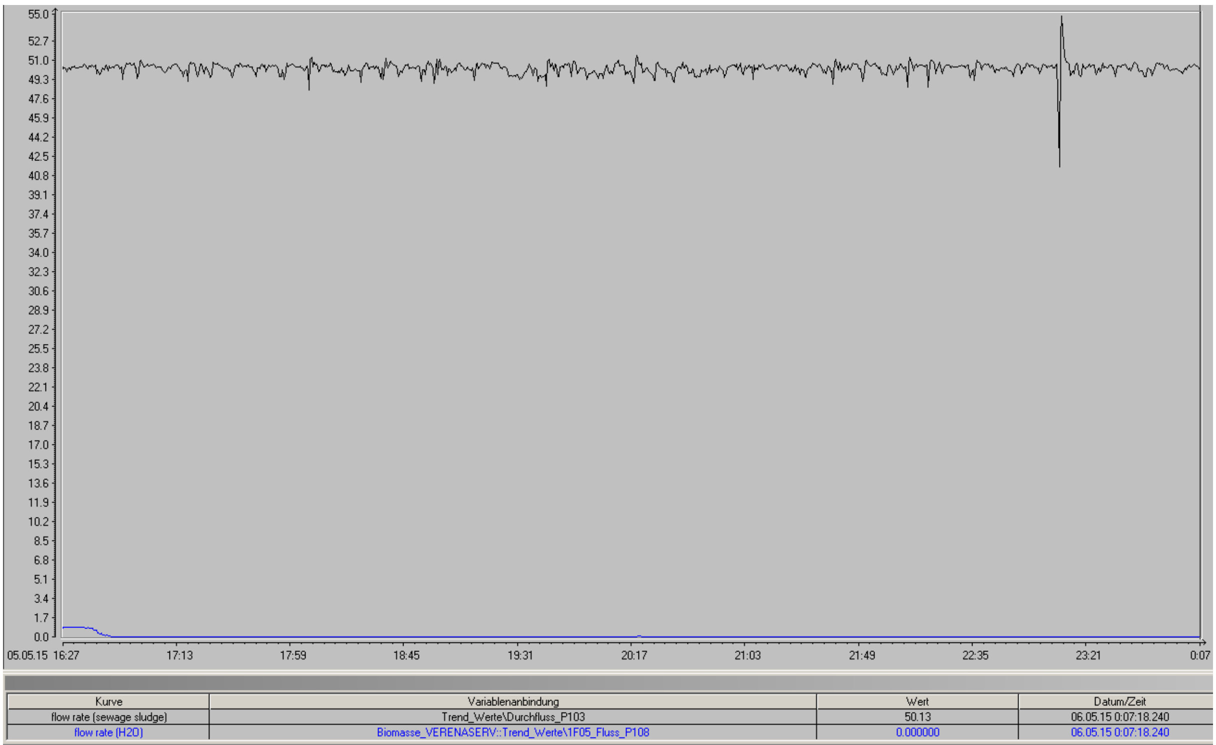


Figure 21: Feed stream of the VERENA test in kg/h during steady state phase.

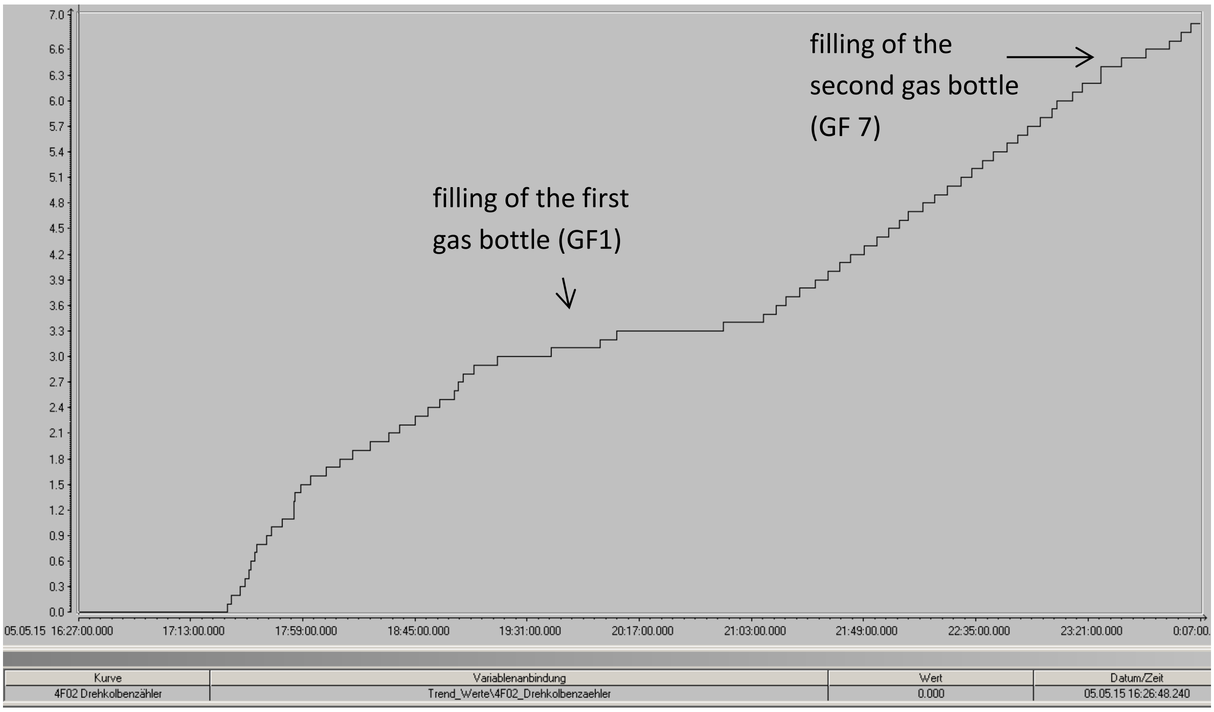


Figure 22: Amount of product gas in Nm^3/h during steady state phase.

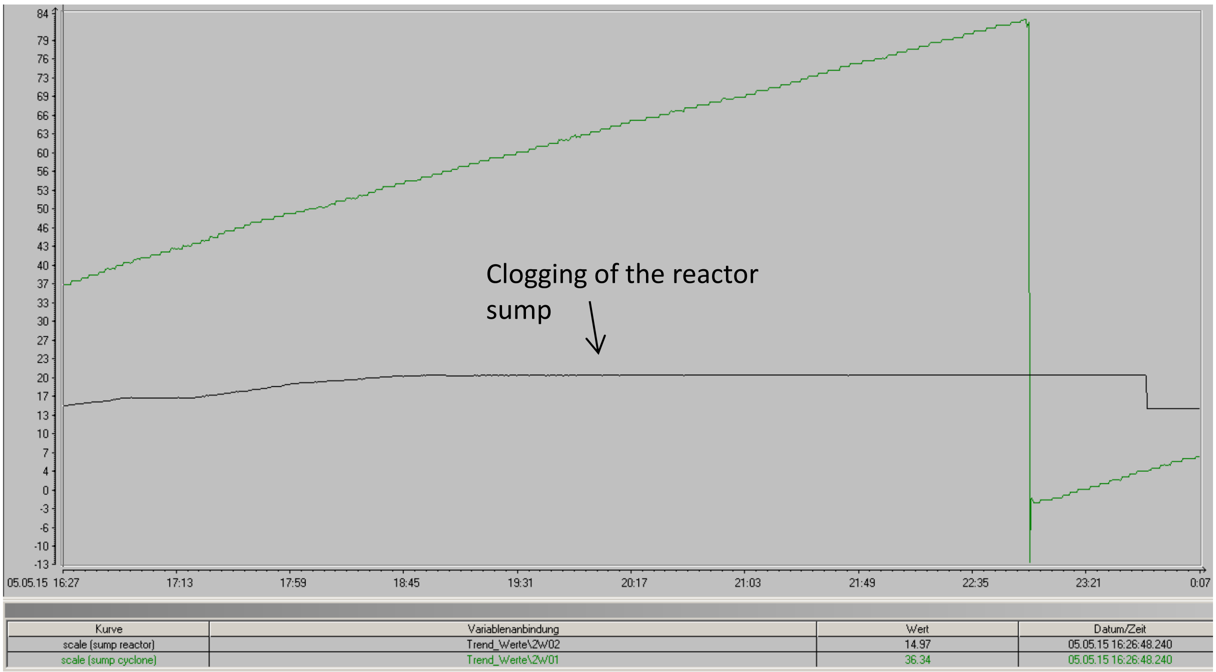


Figure 23: Sum of the salt concentrate separated from the sump of the cyclone (green) and from the sump of the reactor (black) in kg during steady state phase.

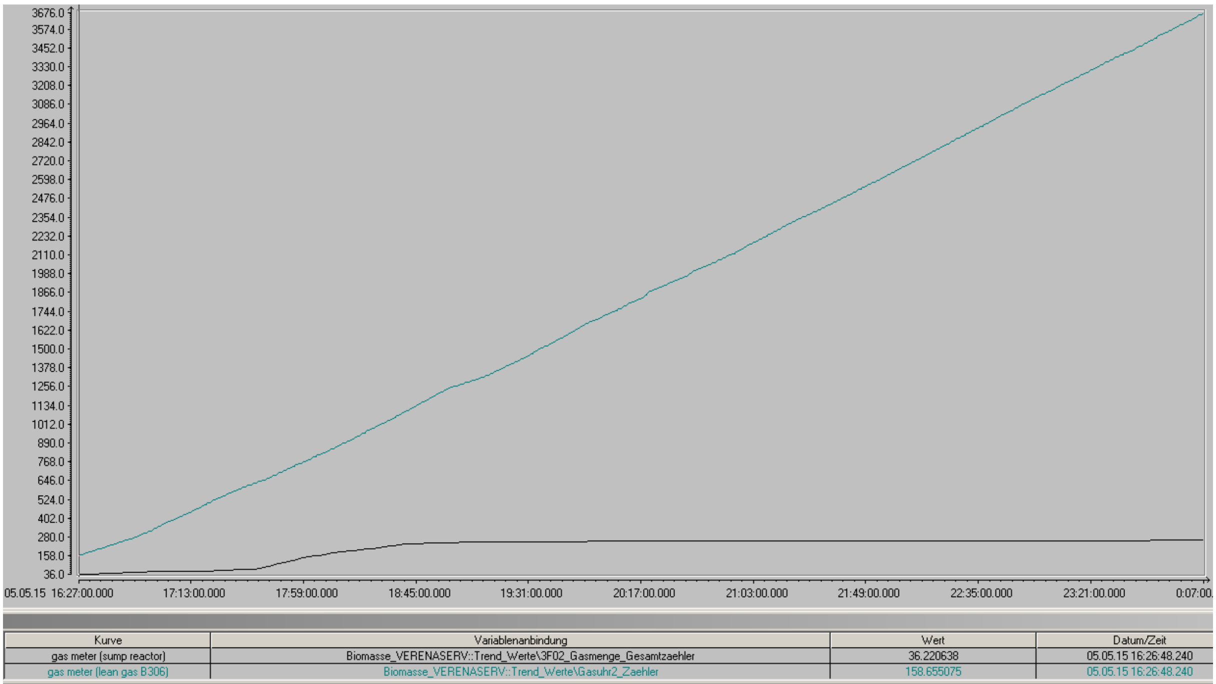


Figure 24: Sum of the lean gas from the waste water (turquois) and the salt concentrate separated from the sump of the reactor (black) in liter during steady state phase.

6.3.3 Experimental results

In the following first the results of the steady state phase are presented. Subsequently the results of the total balance are given.

Experiment conditions		
		Unit
Dry matter content	7,49	%
K+-conc. ($KHCO_3$)	2500	mg/l
mean reactor temperature	640	°C
pressure	270	bar
mean residence time	2,6	min
mean Feed mass flow	50	kg/h

Sewage sludge		
Time	7,67	h
Total sludge fed	383,3	kg
C- mass in Feed	11,8	kg
TOC in Feed	3,1	%
Dry matter	7,49	wt.-%
Total mass effluent	308,7	kg
Total mass salt concentrate liquid	42,1	kg
Total mass salt concentrate solid	12,9	kg
Total mass sump reactor	5,4	kg
Total mass total gas	15,4	kg
TOC-concentration		
in the effluent	1278	mg/kg
in the Salt conc. cyclone	11441	mg/kg
in the Salt conc. reactor	1278	mg/kg
Gas production – product gas	1,30	Nm^3/h
Gas production – total gas	1,86	Nm^3/h
Conversion of gasification	57,1	%
TOC-conversion*	87,0	%
Mass-balance	100	%
C-balance (TC)	77,0	%
N-balance	97,7	%
Product gas composition		
H_2	29,56	Vol %
CO	1,90	Vol %
CO_2	18,32	Vol %
CH_4	33,64	Vol %
C_2H_4	0,82	Vol %
C_2H_6	14,1	Vol %
C_3H_6	0,5	Vol %
C_3H_8	1,15	Vol %

*without the C from $KHCO_3$

Table 42: Results of the steady-state phase

No.	Duration of the phase	Amount of Feed	C_{total}	Y_{Gas}	Y_C	TOC-destruc-tion	Cold gas efficiency η	TOC-waste water	NH_4^+-waste water	TNb-waste water
[-]	[h]	[kg]	[kg]	[%]	[%]	[%]	[-]	[mg/l]	[mg/l]	[mg/l]
KS2	7,67	383,3	11,8	57,1	77,0	87,0	0,66	1278	3150	2769

Y_{Gas} = conversion of gasification; Y_C = C-balance (TC)

In Table 43 the gas production during the steady-state phase is listed. This is composed of the lean gas from the effluent and the lean gas from the two salt concentrates, separated at the sump of the cyclone and reactor. Another component of course is the product gas, which is separated in vessel B301. For experiment KS2 the option for CO_2 scrubbing was retained. Nevertheless, the separation vessel B302, equipped with the integrated CO_2 scrubber was partially filled with the detergent TEGDME and used for the step wise regulation of the pressure. It was decided to not make use of the CO_2 scrubber. This means that the detergent was not circulated, but some part of the CO_2 of the product gas, which was led through vessel B302 during the whole experiment, has been dissolved in TEGDME. The amount of CO_2 dissolved in the detergent is also listed in Table 43 and was taken into account by evaluating the experiment.

Table 43: Gas production during steady-state phase in Nm^3/h

lean gas cyclone	0,052
lean gas sump reactor	0,014
lean gas separator	0,427
product gas	1,299
CO_2 dissolved in TEGDME	0,065
total	1,86

Table 44: Results of the analysis of experiment KS2 (steady state phase: 4:27 pm (5^{th} May) to 0:07 am (6^{th} May))

	Feed		aq. effluent (Position 7)				salt concentrate of cyclone (Position 2)				salt concentrate of reactor (Position 4)		
Parameter	Concentration in dry matter	concentration in sewage sludge*	sample AA35 07:00 pm	sample AA36 08:00 pm	sample AA38 10:00 pm	total B306 S30	sample AZ35 07:00 pm	sample AZ36 08:00 pm	sample AZ38 10:00 pm	solid	sample AS35 07:00 pm	sample AS36 08:00 pm	sample AS38 10:00 pm
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	[mg/l]	[mg/l]	[mg/l]
TC	412	30859	3736	3567	3565	2493	12749	12507	13480	118	4584	3952	3668
TIC	n.d.	n.d.	2543	2359	2374	1544	1493	1505	1414	n.d.	2801	2854	2713
TOC	412	30859	1283	1207	1191	949	11256	11002	12066	118	1783	1098	954
H	58	4344	-	-	-	-	-	-	-	22	-	-	-
N	34	2547	-	-	-	-	-	-	-	n.d.	-	-	-
O	-	-	-	-	-	-	-	-	-	-	-	-	-
NO3	-	-	20,9	21,3	21,6	19,4	20,8	20,7	20,8	-	20,9	20,9	n.d.
NO2	-	-	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-	n.d.	n.d.	n.d.
NH4	-	-	3550	3040	3430	3150	2200	2180	2150	-	3010	2960	2940
TNb	-	-	2807	2757	2683	2209	2064	2110	2033	-	2908	2823	2842
P	19,9	1491	n.d.	0,10	n.d.	0,4	868	966	1047	85	52,0	35,0	37,0
PO4	-	-	n.d.	n.d.	n.d.	16,1	2364	2582	2849	-	158	115	128
S	0,95	71,2	3,90	1,50	1,90	6	27,5	26,0	31,5	0,7	1,90	3,00	31,5
SO4	-	-	16,1	16,4	15,8	15,6	679	663	714	-	33,8	35,0	35,1
Ca	31,7	2374	1,47	0,26	0,53	n.d.	6,65	3,1	4,75	125	2	1,65	3,30
K	45,5	3408	14,6	8,70	6,50	10	11298	2437	2608	70	3530	4035	3845
Mg	21,6	1618	0,9	0,16	0,2	n.d.	75	98,5	95	65	7,50	13,1	6,10
Na	4,1	307	16,6	6,60	5,40	2,2	1012	1083	1100	7	269	308	293
Si	35	2622	16,3	4,90	3,90	n.d.	34,5	34	32	101	0,21	n.d.	1,55
Al	6,8	509	0,16	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	38	54,0	62,1	60,6
As	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cu	1,1	82,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,00	n.d.	n.d.	n.d.
Pb	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Zn	1,26	94,4	0,04	n.d.	n.d.	n.d.	1,2	0,6	1,1	6,00	0,21	n.d.	1,55
Cr	0,034	2,5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1,10	n.d.	n.d.	n.d.

	Feed		aq. effluent (Position 7)				salt concentrate of cyclone (Position 2)				salt concentrate of reactor (Position 4)		
Parameter	Concentration in dry matter	concentration in sewage sludge*	sample AA35 07:00 pm	sample AA36 08:00 pm	sample AA38 10:00 pm	total B306 S30	sample AZ35 07:00 pm	sample AZ36 08:00 pm	sample AZ38 10:00 pm	solid	sample AS35 07:00 pm	sample AS36 08:00 pm	sample AS38 10:00 pm
[-]	[mg/g]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/g]	[mg/l]	[mg/l]	[mg/l]
Fe	24,3	1820	n.d.	n.d.	n.d.	n.d.	3,7	17	8,3	95	0,78	n.d.	n.d.
Mo	0,011	0,8	7,7	3	0,6	0,7	288	263	345	1,7	3140	3885	3795
Ni	0,017	1,3	n.d.	n.d.	n.d.	0,9	n.d.	n.d.	n.d.	18	61	240	461
Cl	3,78	283	n.d.	n.d.	n.d.	25,1	1471	1527	1724	2,3	115	128	127
Hg	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Phenolindex	-	-	270	247	224	183	140	140	141	-	192	173	184

* for simplification it is assumed that the density of water equates to the density of sewage sludge

Legend:	n.d.	not detected

		Feed	cyclone solid	
Ash content	by 550°C:	24,29	82,4	%
	by 815°C:	23,58	81,8	%
	by 1000°C:	23,26	80,8	%
Higher Heating Value:		17202,3	-	J/g

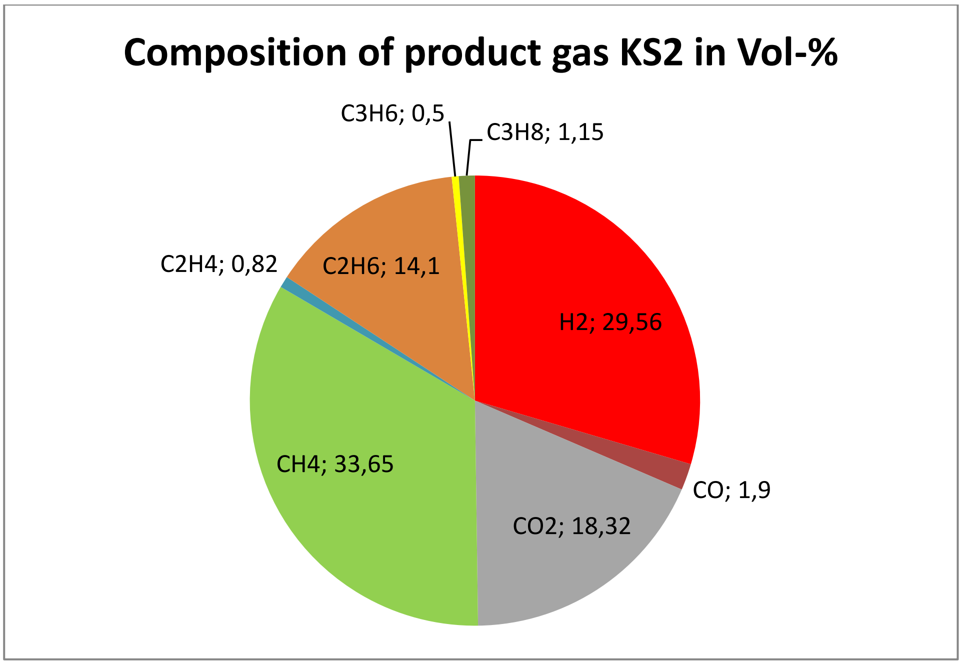


Figure 25: Composition of the product gas of experiment KS2.

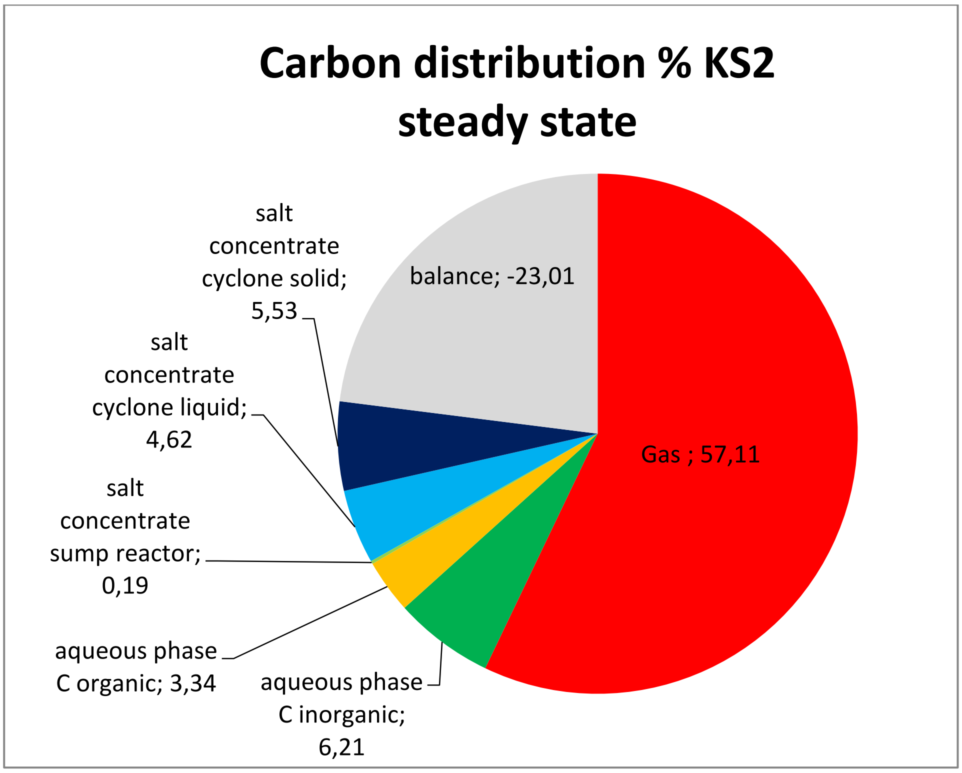


Figure 26: Carbon distribution of experiment KS2 during the steady state phase.

Table 45: composition of the lean gas and product gas of experiment KS2.

date	time	x_{H2} [vol%]	x_{CO} [vol%]	x_{CH4} [vol%]	x_{CO2} [vol%]	x_{C2H4} [vol%]	x_{C2H6} [vol%]	x_{C3H6} [vol%]	x_{C3H8} vol%]	sum	Kommentar
lean gas sump cyclone B201											
05.05.2015	07:00	36,62	2,84	14,32	41,08	1,62	3,51	-	-	100,00	
05.05.2015	17:40	40,83	2,40	9,35	43,09	1,23	1,45	1,09	0,57	100,00	
05.05.2015	19:40	38,34	2,13	10,02	45,49	1,12	1,23	1,11	0,56	100,00	nitrogen was not measured
05.05.2015	21:40	38,55	1,99	10,17	45,99	0,93	0,89	1,03	0,45	100,00	
05.05.2015	23:40	37,68	1,90	10,55	46,79	0,89	0,77	1,00	0,41	100,00	
lean gas effluent B306											
05.05.2015	06:00	14,44	0,00	11,71	70,86	0,43	2,44	0,06	0,05	100,00	
05.05.2015	18:40	14,05	0,00	10,39	73,16	0,27	2,02	0,05	0,05	100,00	
05.05.2015	20:40	12,85	0,00	10,02	74,66	0,32	2,03	0,07	0,06	100,00	nitrogen was not measured
05.05.2015	22:40	13,89	0,00	10,23	73,51	0,31	1,97	0,05	0,05	100,00	
06.05.2015	00:40	15,21	0,00	10,43	71,77	0,28	2,20	0,06	0,06	100,00	
lean gas sump reactor B203											
05.05.2015	07:00	28,98	1,72	26,05	31,40	0,63	10,26	0,43	0,54	100,00	
05.05.2015	17:40	31,37	1,23	25,87	29,40	0,57	10,45	0,40	0,71	100,00	
05.05.2015	19:40	24,50	1,65	28,48	32,57	0,51	11,47	0,00	0,82	100,00	nitrogen and oxygen are deducted
05.05.2015	21:40	24,70	1,69	29,36	31,56	0,50	11,42	0,00	0,77	100,00	
05.05.2015	23:40	24,36	1,33	28,88	32,45	0,51	11,66	0,00	0,82	100,00	
product gas B301											
05.05.2015	06:00	30,73	2,35	34,86	12,92	1,17	16,12	0,73	1,12	100,00	
05.05.2015	16:40	30,87	2,52	34,28	13,63	1,19	15,45	0,81	1,25	100,00	
05.05.2015	18:40	30,65	2,05	32,74	17,64	0,87	14,27	0,55	1,24	100,00	nitrogen is deducted
05.05.2015	20:40	28,89	1,69	34,32	18,35	0,84	14,27	0,50	1,13	100,00	
05.05.2015	22:40	29,15	1,95	33,87	18,97	0,76	13,77	0,45	1,08	100,00	
06.05.2015	00:40	26,98	1,98	33,67	21,30	0,72	13,86	0,42	1,07	100,00	
product gas from the gas bottles											
GF1		28,38	2,01	33,48	18,96	0,84	14,58	0,53	1,22	100,00	nitrogen and oxygen are deducted
GF7		28,66	2,00	32,67	20,26	0,75	14,01	0,48	1,17	100,00	

Table 46: Lean gas of the salt concentrate separated from the sump of the cyclone during steady state phase (offline data).

date	time	cyclone gas meter / NL
05.05.15	16:00 - 17:00	37,2
05.05.15	18:00	63,24
05.05.15	19:00	73,47
05.05.15	20:00	55,8
05.05.15	21:00	53,01
05.05.15	22:00	48,36
05.05.15	23:00	53,01

Besides the balance during the steady state operation - which reflects the operation of a commercial plant working all around the clock - a second kind of balance called here as total balance (as mentioned before) can be performed. Some additional information about the distribution of the inorganic salts can be gained. During lab-scale operation, a better balance has also been achieved. This is only the case if all aqueous solution used to clean the plant are measured and analyzed. Since this is not practical for the large pilot plant, the informative value of the "total balance" for the two pilot plant experiments is low. Nevertheless, the total balance is presented here to complete the picture.

Table 47: total masses of all in and out streams of the VERENA plant during experiment KS2 (from the beginning of the first feeding of sewage sludge till the end of the experiment)

evaluation period for total balance	46,1 h		2:17 pm 04.05.15 - 12:25 pm 06.05.15		Only for B306 balance 4:27 pm 05.05.15 - 00:07 am 06.05.15	
Feed						
DM Feed			7,49	wt.-%		
Feed total			598100	g	598100	g
H_2O total			2215000	g		
effluent and salt concentrate						
total effluent (calculated!)			2411976	g	503840	g
salt concentrate:						
liquid sump reactor			92700	g		
liquid sump cyclone			313450	g		
solid sump cyclone	wet (42,9 wt.-%DM)		22850	g		
total sump cyclone			336300	g		
total gas			24700	g		
Gasphase	**C-amount**		**gas amount:**			
B306 (measured by a gas meter)	3848	g C	12665	g		
B203 (measured by a gas meter)	188	g C	417	g		
B201 (measured by a gas meter)	154	g C	472	g		
product gas (rotary meter and gas bottles)	5473	g C	10156	g		
CO_2 TEGDME	**270**	g C	**990**	g CO_2		
total:	**9933**	g C	**24700**	g		

Notice: The vessel B306 does not represent a cumulative sample of the whole process like the other vessels, but is a throughput vessel with a long residence time of about 1 to 2 hours. So balance of the aqueous phase is performed for the period of steady state operation.

The amount of solids in the salt concentrate from the sump of the cyclone was only determined for the steady state phase. Therefore for the total balance the amount of solids in this salt concentrate was adjusted to the total amount of feed pumped into the plant during KS2.

In Table 48 and Table 49 the element balance (total) is listed.

Table 48: element balance of non-metal elements.

Element	N		P		S		Cl		Si	
	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %
Feed	1523,12		891,47		42,56		169,34		1567,92	
aqueous phase	1112,98	73,07	0,20	0,02	3,02	7,10	12,64	7,46	0,00	0,00
salt concentrate sump reactor	127,59	8,38	4,34	0,49	1,32	3,11	10,42	6,16	0,00	0,00
salt concentrate cyclone liquid	238,85	15,68	118,69	13,31	16,08	37,78	99,06	58,50	0,00	0,00
salt concentrate cyclone solid	0,00	0,00	833,03	93,44	6,86	16,12	22,54	13,31	989,84	63,13
total	1479,42	97,13	956,26	107,27	27,28	64,11	144,66	85,43	989,84	63,13

Table 49: element balance of metal elements.

Element	K		Ca		Mg		Fe		Al	
	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %
Feed	3211,20		1420,09		967,63		1088,58		304,62	
aqueous phase	5,04	0,16	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
salt concentrate sump reactor	234,91	7,32	0,00	0,00	16,59	1,71	0,02	0,00	0,00	0,00
salt concentrate cyclone liquid	733,09	22,83	0,00	0,00	14,29	1,48	0,04	0,00	0,00	0,00
salt concentrate cyclone solid	686,03	21,36	1225,05	86,27	637,03	65,83	931,04	85,53	372,42	122,25
total	1659,07	51,67	1225,05	86,27	667,90	69,02	931,10	85,53	372,42	122,25

Table 50: C-balance of KS2 (total balance)

Element	C	
	Mass, g	Recovery, %
Feed	18456,65	
Gas	9933	53,82
aqueous phase C organic	478,14	2,59
aqueous phase C inorganic	777,93	4,21
salt concentrate sump reactor	186,96	1,01
salt concentrate cyclone liquid	1410,93	7,64
salt concentrate cyclone solid	1156,45	6,27
total	13943,41	75,55

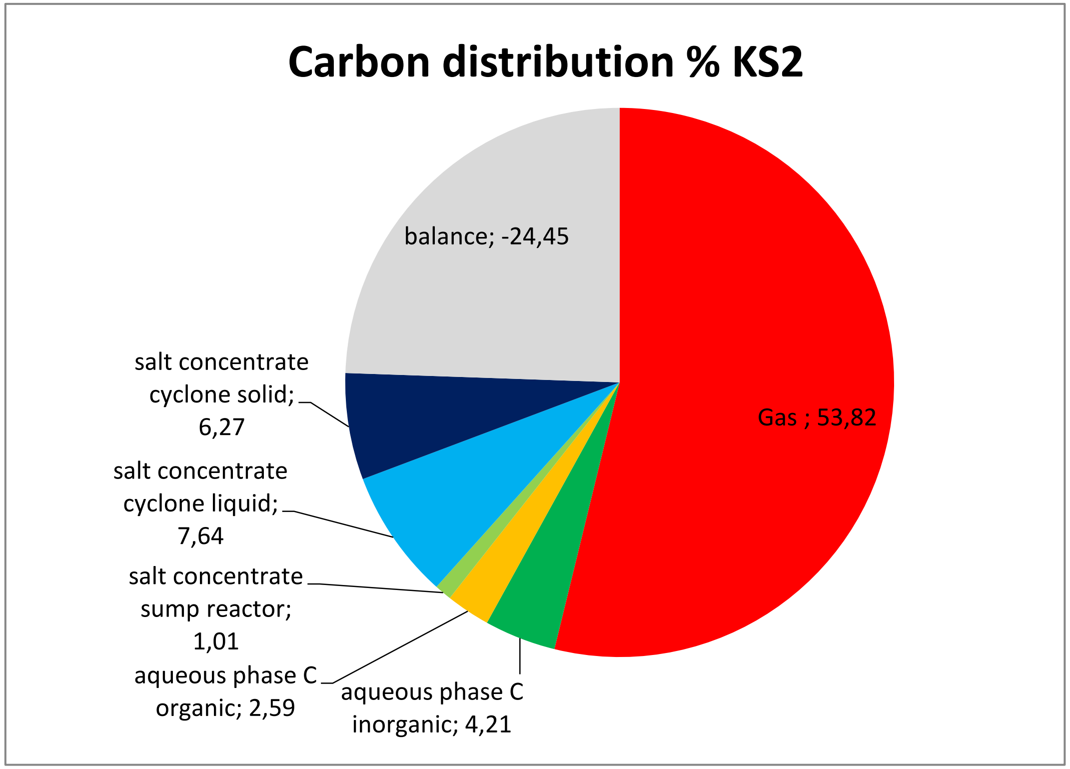
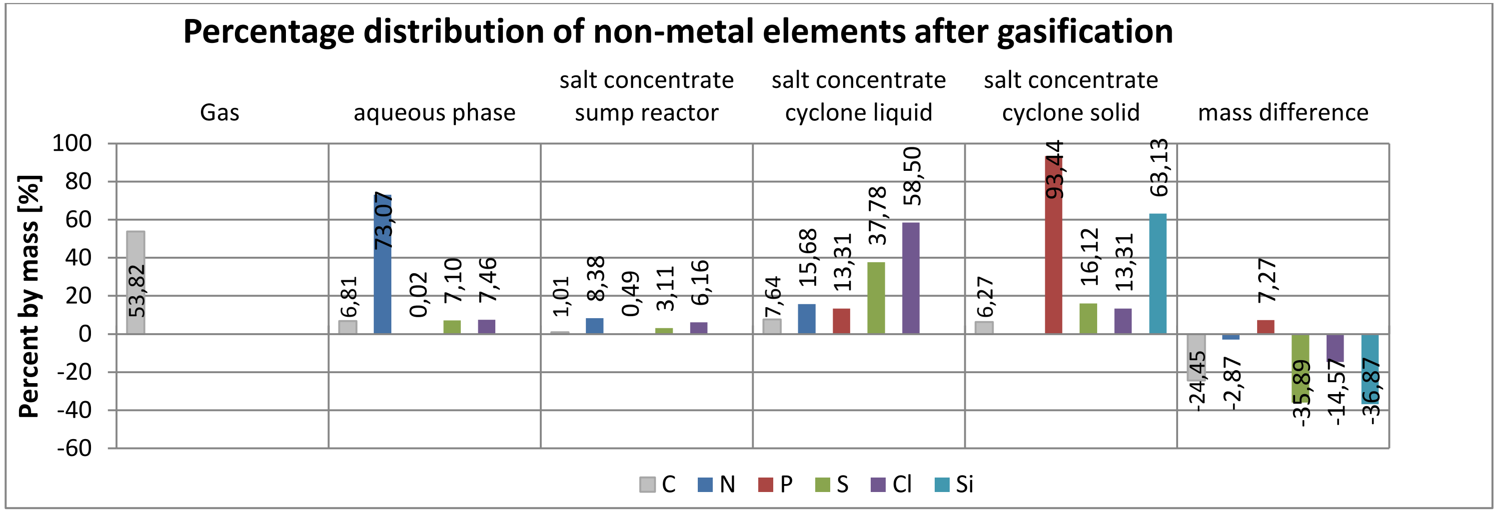
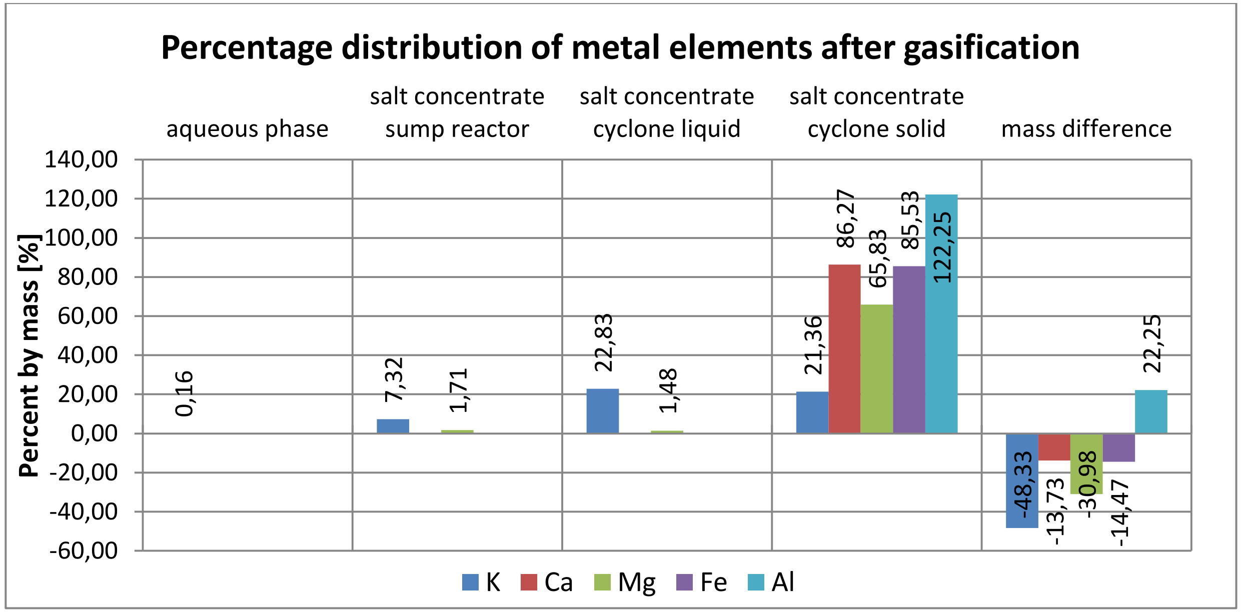


Table 51: Comparison of conversion of gasification and TOC-conversion of the total balance and during steady state operation

	conversion of gasification [%]	TOC-conversion [%]
total balance	**53,8**	**84,2**
steady state operation	**57,1**	**87,0**


Percentage distribution of non-metal elements after gasification
Gas
aqueous phase
salt concentrate sump reactor
salt concentrate cyclone liquid
salt concentrate cyclone solid
mass difference
Percent by mass [%]
100
80
60
40
20
0
-20
-40
-60
53,82
6,81
73,07
0,02
7,10
7,46
1,01
8,38
0,49
3,11
6,16
7,64
15,68
13,31
37,78
58,50
6,27
93,44
16,12
13,31
63,13
-24,45
-2,87
7,27
-35,89
-14,57
-36,87
C N P S Cl Si


Percentage distribution of metal elements after gasification
aqueous phase
salt concentrate sump reactor
salt concentrate cyclone liquid
salt concentrate cyclone solid
mass difference
Percent by mass [%]
140,00
120,00
100,00
80,00
60,00
40,00
20,00
0,00
-20,00
-40,00
-60,00
0,16
7,32
1,71
22,83
1,48
21,36
86,27
65,83
85,53
122,25
-48,33
-13,73
-30,98
-14,47
22,25
K Ca Mg Fe Al

In Table 52 the balance of minor elements is shown. The concentrations of As, Pb, Cd, Hg in the feed are under 0,01 wt.-%. The limit of quantification of liquids is dependent from the dilution factor. Since the liquid samples often contain a lot of salts and organics the samples also have to be diluted for minor analysis. Therefor the limits of quantification (see Appendix) are accordingly high. The data measured by Aquon are listed in the Appendix.

The increased Mo, Ni and Cr-values indicate corrosion.

Table 52: balance of minor elements

Element	Cu		Zn		Na		Mo		Ni		Cr	
	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery, %	Mass, g	Recovery , %
Feed	49,28		56,45		183,67		0,49		0,76		1,52	
aqueous phase	0,00	0,00	0,00	0,00	1,11	0,60	0,35	71,57	0,45	59,09	0,00	0,00
salt concentrate sump reactor	0,00	0,00	0,00	0,00	14,67	7,99	208,72	42356,08	11,58	1520,56	0,00	0,00
salt concentrate cyclone liquid	0,00	0,00	0,00	0,00	90,00	49,00	27,40	5560,35	0,10	13,39	0,00	0,00
salt concentrate cyclone solid	49,00	99,44	58,80	104,18	68,60	37,35	16,60	3380,86	176,41	23164,27	10,78	707,76
total	49,00	99,44	58,80	104,18	174,38	94,94	253,13	51368,86	188,54	21757,32	10,78	707,76

In Table 53 the ash content of the feed and the salt concentrate separated from the sump of the cyclone are listed. 54,48 % of the ash content of the feed were separated by the cyclone. No ash separation from the reactor and the waste water was performed.

Table 53: Ash content of the Feed and the salt concentrate separated from the sump of the cyclone.

Ash in total Feed	10419,94	g
Ash in salt concentrate cyclone	7918,73	g
Ash salt concentrate/Ash Feed	76,00	%

Additional analysis of the product gas

Table 54: results of the tar measurement of the product gas (first measurement).

20 ml Wasser/Ethylenglykol 9:1 2 °C, 30 min, ~ 1,5 Nl	Blind, 19.KW		Blind, 19.KW		VERENA , 19.KW		VERENA, 19.KW		
1. Messung (08.05.15)	S21 A		S21 B		S28 A		S28 B		A+B
	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	mg/Nm3
Benzene	0,73	0,5	3,47	2,3	3194	2129	2468	1645	3774
Toluene	6,20	4,1	8,17	5,4	1035	690	217	145	835
Naphthalene	63,2	42,1	67,0	44,7	238	159	149	99	258

Table 55: results of the tar measurement of the product gas (second measurement).

20 ml Wasser/Ethylenglykol 9:1 2 °C, 30 min, ~ 1,5 Nl	Blind, 19.KW		Blind, 19.KW		VERENA , 19.KW		VERENA, 19.KW		
2. Messung(11.05.15)	S21 A		S21 B		S28 A		S28 B		A+B
	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	µg/Probe	mg/Nm3	mg/Nm3
Benzene	0,60	0,4	0,82	0,6	2145	1430	1665	1110	2540
Toluene	4,11	2,7	3,67	2,4	842	562	154	102	664
Naphthalene	64,1	42,7	96,0	64,0	225	150	163	108	268

The H_2S and NH_3 measurement of the product gas showed the following concentrations: 0,0016-0,0044 vol% H_2S and <0,0001 vol% NH_3.

	B301 05.05.15 21:15	B301 05.05.15 23:20	product gas, GF1	product gas, GF7
	Vol%	Vol%	Vol%	Vol%
H_2S	0,0022	0,0043	0,0016	0,0044
NH_3	< 0,0001	< 0,0001	< 0,0001	< 0,0001

In Table 56 the Hg-measurements done by Tauw B.V. according to NEN-EN 13211: 2001 Norm are listed. The amount of Hg (0,003 mg/Nm^3) in the product gas is very low.

Table 56: results of the Hg measurement of the product gas from gas bottle done by Tauw B.V.

Results measurements Mercury Karlsruher Instiut fur Technologie, bottle

Parameter	eenheid	measurement 1	measurement 2	measurement 3	average
date	[dd-mm-yyyy]	06-05-2015	06-05-2015	06-05-2015	
time start	[hh:mm]	13:40	14:12	14:44	
time stop	[hh:mm]	14:10	14:42	15:14	
mercury	[mg/Nm³]	0,002	0,003	0,003	0,003

11 Conclusions and recommendation

The present project includes three parts. Several experiments in the Lab-scale and two experiments in the pilot scale.

As first part of this project several lab scale experiments were carried out. These experiments comprised the gasification of sewage sludge from the municipal purification plants of Lelystad and Oijen and were performed in a dedicated continuous flow test rig. It was demonstrated that the gasification of sewage sludge in supercritical water is feasible. Stable operation is possible, even in small delicate lab scale equipment and with concentrated sludge (up to 17 wt. % DM). The operation proceeded better than with plant biomass, which contains bigger particles and is less homogeneous than the sewage sludge. After optimization of the process conditions and adaption of the experimental procedures to the sewage sludge good carbon balances (100 ± 10 %) were reached. High gasification yields of up to 80 % were achieved while the process has a cold gas efficiency of up to 0.95. The highest cold gas efficiency was observed by gasification of a feed with a concentration of 13 wt. % DM. During the experiments with both sludges the salts could be separated by about 50 %. Several minor elements (heavy metals) could not be detected and therefore could not be balanced. This only accounts for amounts less than 1 g of a total experiment. Altogether a compressed, clean product gas with high calorific value in one process step can be gained. During experiments with the Lab scale plant no severe corrosion has been observed. This is due to the alloys used in the different parts of the plant, especially alloy 602 as reactor material. Only after all the experiments with sewage sludge and some additional gasification experiments with micro algae, a lower temperature tube made of stainless steel 316 leaked.

During this set of experiments some solid material was detected after disassembling of lines (tubes) not in the reactor. Although in some cases coke – soot were detected, the total amounts were low. The same is the case for corrosion products. Tar was found in an amount of few g in the filter cake of the waste water phase.

As second part of this project a first experiment in the pilot scale was carried out. This experiment was the first gasification of sewage sludge from the municipal purification plant of Oijen in the pilot scale and was performed in the dedicated continuous flow pilot plant VERENA.

The VERENA plant, previously improved with new pre-heaters, performed well and reached the desired reaction temperature of 660 °C.

It was demonstrated that the gasification of sewage sludge (11,8 wt%) in supercritical water with a representative amount of 400 kg sewage sludge is feasible. For the first time effective (but not complete) separation of the inorganic salts of the feed was realized. The TOC of the feed stream has been reduced by more than 85 %. The only solid matter exiting the process is brine containing inorganic salts (phosphates and carbonates).

A lot of new knowledge has been gained from this experiment, valuable for the engineering of a commercial plant:

This experiment showed that the product gas contained H_2S, 0.1 vol %, a high value.
Tar concentration in the product streams (both aqueous and gaseous) is an issue for the quality of the product gas, the quality of the waste water and the stability of operation. The gasification efficiency was lower than in an experiment in the lab scale under similar conditions.
During experiment an increase of the pressure difference inlet – outlet has been observed, a clear indication of fouling. Also the viscosity of the feed was high for the tubing used in the VERENA plant (8 mm i.d.).
Erosion of some parts of the system for the salt separation was high.
The first experiment ended with a very persistent plug in the main pre-heater of the plant.
As a third part of this project, a second experiment in the pilot-scale plant was carried out. This was the second gasification experiment of sewage sludge from the municipal treatment plant of Oijen in the pilot scale and it was performed –as the first experiment- in the dedicated continuous flow pilot plant VERENA. In order to avoid plugging of the plant and gain better results compared to the first experiment, the sludge had a lower concentration of about 7 wt. % DM.
After the first experiment and the repair of the pre-heater, a new campaign was executed in April 2015. During start-up operation with water the plant showed again clogging. The plug was removed but after discussion with the Dutch delegation present the feeding of sewage sludge did not start because of the uncertainties concerning the solids in the reaction system.
Then, the sewage sludge prepared for the experiment was disposed. The plant was thoroughly cleaned by one week operation with water at the operation conditions (T>600 °C, p=280 bar) and additionally by gasification of an ethanol solution for several (40) hours. After this campaign, parts of the plant tubing were disassembled and cleaned. Dark solid matter from the first experiment with sewage sludge has been removed. Experiment with new sewage sludge has been performed with a third campaign in May 2015.
This experiment showed smooth steady state operation during several (8) hours. Two high pressure bottles were filled with the product gas. Several samples have been collected and were analysed.
The minor elements in the feed are balanced better than 80 %. Only K, Mg, S and Si show a poorer balance (mostly 60 %). Also Ni, Mo and Cr show a much higher amount in the product streams than in the feed. This is a clear evidence of corrosion of the reaction system.
The separation of salts was improved and 76 % for the total ash was found in the cyclone concentrate. Phosphorus was recovered in the cyclone concentrate.
The gasification yield was 57 % a value which is lower compared to the lab-scale experiments. About 4 % of the carbon forms after reaction inorganic soluble components most probably NH_4HCO_3. Some 3 % of the Carbon contained in the feed forms the TOC of the waste water and 14 % is found in the cyclone concentrate.
There is a difference on the feed composition between the lab scale and the pilot scale experiments: The concentrated sewage sludge was diluted with pure water for the lab scale experiments. The feed for pilot plant experiments was diluted with the water phase gained

from the dewatering of the sewage sludge. Thus there were additional not identified organic and inorganic substances present.
Also during this experiment tar formation was observed. The experiment was interrupted due to indications of beginning plug in the high pressure system. During restart pressure drop occurred. This conducted the end of the experiment.
The leak was in the pre-heater.
After disassembling the reactor solid corrosion products were collected from the lower part of the reactor. The reactor contained several different materials and so it is not easy to localize exactly the corrosion.

- The material alloy 625 should be avoided for the construction of a commercial plant. Mechanical stress during fabrication of the components and tubing should be minimized, especially in the high temperature part of the plant. This is due to dealloying of Mo from alloy 625 during operation.

- Tar concentration in the product streams (both aqueous and gaseous) is an issue for the quality of the product gas and waste water. The gasification efficiency was lower compared to the experiments in the lab scale under similar conditions. The stream with salt concentrate contained, due to the temperature of 470 °C, tarry material. If the process layout provided salt separation at temperatures higher than 600 °C only low tarry material would be found. A salt separation after the reaction zone would result in a much lower concentration of organics in the salt concentrate. This can be realized only with other process layout –like the so called two stream feeding in the VERENA which is more expensive- or with other hardware.

- Erosion of the system for the salt separation was high. To mitigate erosion, a further improvement in the salt separation system is necessary.

- Additional experiments, both in the lab eventually with other process layout and hardware and in the pilot scale, are recommended in order to understand the reduced gasification efficiency during scale up to the pilot plant scale.

12 Acknowledgements

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13 Literature

Acelas, Nancy Y.; López, Diana P.; Brilman, D.W.F.; Kersten, Sascha R.A.; Kootstra, A. Maarten J. (2014): Supercritical water gasification of sewage sludge: Gas production and phosphorus recovery. In: *Bioresource Technology* 174, S. 167–175. DOI: 10.1016/j.biortech.2014.10.003.

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Gong, M.; Zhu, W.; Xu, Z. R.; Zhang, H. W.; Yang, H. P. (2014): Influence of sludge properties on the direct gasification of dewatered sewage sludge in supercritical water. In: *Renewable Energy* 66, S. 605–611. DOI: 10.1016/j.renene.2014.01.006.

Data from VERENA plant experiments with corn silage are published here:

N. Boukis, U. Galla, P. D´Jesus, H. Müller and E. Dinjus,
Gasification of Wet Biomass in Supercritical Water. Results of Pilot Plant Experiments
14th European Conference on Biomass for Energy, Industrie and Climate protection, 17 - 21 October 2005, Palais des Congrès, Paris, France, Proceedings 964-967

Appendix

Results of analysis by Aquon on samples from STOWA VERENA test run #1

Component	Unit	sewage sludge *calculated values for dilution*	AZ (B202) salt from cyclone	% of feed	AS (B204) salt from reactor	% of feed	Auslauf (B306) effluent	% of feed	total% recovered
BOD 5 days (allylthioureum)	mg O2/l	38500	2110	5%	1320	3%	1550	4%	13%
COD	mg O2/l	159366	5520	3%	5710	4%	5320	3%	10%
BOD/COD ratio	-	0,24	0,38		0,23		0,29		
Kjeldahl-N	mg N/l	5967	370	6%	1900	32%	4700	79%	117%
Ammonium	mg N/l	1638	150	9%	1600	98%	3600	220%	327%
Cl, dissolved	mg/l	702	450	64%	69	10%	140	20%	94%
P total	mg P/l	2340	250	11%	190	8%	3,1	0%	19%
S total	mg S/l	573	84	15%	8,7	2%	17	3%	19%
metals									
Mg	mg/l	<293	43		38		11		
Al	mg/l	959	120	13%	110	11%	0,52	0%	24%
K	mg/l	<1170	1100		330		48		
Ca	mg/l	<1170	160		160		25		
Cr	ug/l	3744	660	18%	490	13%	<50		31%
Fe	mg/l	2106	160	8%	160	8%	<1,5		15%
Ni	ug/l	2223	9000	405%	16000	720%	1300	58%	1183%
Cu	ug/l	44460	4100	9%	1600	4%	120	0%	13%
Zn	ug/l	83070	9300	11%	7600	9%	120	0%	20%
As	ug/l	410	<25		<25		<25		
Mo	ug/l	1112	2500	225%	5600	504%	27	2%	731%
Cd	ug/l	103	<20		<40		<5		
Sb	ug/l	433	10	2%	<5		<5		2%
Pb	ug/l	7722	640	8%	460	6%	<10		14%
Hg	ug/l	53	<0,2		3,4	6%	2,1	4%	10%
PAH 16 components (EPA)	µg/l		0,72		123016		87057		
PAH 10 comptontents (VROM)	µg/l	620	0,58	0%	109171	17605%	73394	11836%	29441%
Naphtalene	µg/l	<6	0,46		98316		59123		
Acenaphthylene	µg/l	<6	<0,04		1728		1779		
Acenaphthene	µg/l	<6	0,04		4126		3295		
Fluorene	µg/l	<6	0,05		6372		5792		
Phenanthrene	µg/l	63	0,06		7565		8197		
Anthracene	µg/l	9	<0,01		1958		2541		
Fluoranthene	µg/l	176	<0,01		907		1684		
Pyrene	µg/l	<6	<0,02		1556		2581		
Benz[a]anthracene	µg/l	69	0,02		113		444		
Chrysene	µg/l	75	<0,01		188		788		
Benzo[b]fluoranthene	µg/l	<6	<0,01		60		188		
Benzo[k]fluoranthene	µg/l	40	<0,01		20		104		
Benzo[a]pyrene	µg/l	70	<0,01		59		312		
Dibenz[a,h]anthracene	µg/l	<6	<0,01		3,4		28		
Benzo[ghi]perylene	µg/l	54	<0,01		23		100		
Indeno[1,2,3-cd]pyrene	µg/l	64	<0,01		22		102		
Inhibition of biological nitrification compared to reference									
No dilution	%						100%		
5x dilution	%						100%		
10x dilution	%						100%		
50x dilution	%						100%		

Component	Unit	Sewage sludge
Dry matter content	% dry matter	11,7%
Ash residu	% of dry matter	22%
BOD 5 days (allylthioureum)	mg O2/l	38500
COD	mg O2/ kg dry matter	1362100
Kjeldahl-N	g N/kg dry matter	51
Ammonium	g N/kg dry matter	14
Cl, dissolved	mg/kg dry matter	6000
P total	g P/kg dry matter	20
S total	g S/kg dry matter	4,9
metals		
Mg	mg/kg dry matter	<2500
Al	mg/kg dry matter	8200
K	mg/kg dry matter	<10000
Ca	mg/kg dry matter	<10000
Cr	mg/kg dry matter	32
Fe	mg/kg dry matter	18000
Ni	mg/kg dry matter	19
Cu	mg/kg dry matter	380
Zn	mg/kg dry matter	710
As	mg/kg dry matter	3,5
Mo	mg/kg dry matter	9,5
Cd	mg/kg dry matter	0,88
Sb	mg/kg dry matter	3,7
Pb	mg/kg dry matter	66
Hg	mg/kg dry matter	0,45
PAH 16 components (EPA)	mg/kg dry matter	
PAH 10 comptontents (VROM	mg/kg dry matter	5,3
Naphtalene	mg/kg dry matter	<0,05
Acenaphthylene	mg/kg dry matter	<0,05
Acenaphthene	mg/kg dry matter	<0,05
Fluorene	mg/kg dry matter	<0,05
Phenanthrene	mg/kg dry matter	0,54
Anthracene	mg/kg dry matter	0,08
Fluoranthene	mg/kg dry matter	1,5
Pyrene	mg/kg dry matter	<0,05
Benz[a]anthracene	mg/kg dry matter	0,59
Chrysene	mg/kg dry matter	0,64
Benzo[b]fluoranthene	mg/kg dry matter	<0,05
Benzo[k]fluoranthene	mg/kg dry matter	0,34
Benzo[a]pyrene	mg/kg dry matter	0,6
Dibenz[a,h]anthracene	mg/kg dry matter	<0,05
Benzo[ghi]perylene	mg/kg dry matter	0,46
Indeno[1,2,3-cd]pyrene	mg/kg dry matter	0,55
Inhibition of biological nitrification compared to reference		
No dilution	%	
5x dilution	%	
10x dilution	%	
50x dilution	%	

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ANALYSERAPPORT

Datum	**Code**	**Versie**	**Informatie**
27-05-2015	R150527028	1	klantvragen@aquon.nl

Opdrachtreferentie	2015-351
Opdrachtomschrijving	2015 Voorbereidingen VERENA test KIT WsAM
Opdracht referentie klant	5.5220.054
Rapportage ontvanger	P. van Vugt

Geachte heer van der Molen,

Hierbij zend ik u de resultaten van het laboratoriumonderzoek dat op uw verzoek is uitgevoerd. Deze analyses hebben uitsluitend betrekking op de monsters, zoals die door u ter analyse zijn aangeboden.

De werkzaamheden zijn, tenzij anders aangegeven, uitgevoerd conform de PDC van AQUON. Tevens is in het "overzicht methodes AQUON" informatie over de toegepaste onderzoeksmethoden te vinden. Beide documenten zijn te vinden op de website van AQUON: http://www.aquon.nl/downloads/downloads.aspx.
Informatie over uitbestede analyses (prestatiekenmerken, accreditatie, toegepaste normen) kan worden opgevraagd bij accountmanagement van AQUON.

Het analyserapport mag slechts in zijn geheel worden gereproduceerd, tenzij vooraf schriftelijk toestemming van het laboratorium wordt verkregen.

Ik vertrouw erop uw opdracht naar tevredenheid en conform de afspraak te hebben uitgevoerd. Heeft u naar aanleiding van deze rapportage nog vragen, dan verzoek ik u contact op te nemen met het laboratorium via het bovenstaande emailadres.

Hoogachtend,

Ir. E.F.M. Nieuwenhuis
Adjunct-Directeur

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Rapportnummer: R150527028 / 1

Monsternummer	15-038777		Opm.: 1
Monsterpuntcode	302999		
Monsterpuntomschrijving	RWZI Oijen, Diversen		
Matrix	Zuiveringsslib		
Type bemonstering	Steekmonster		
Soort onderzoek	Project		
Contramonster	Nee		
Monsternemingsdatum/tijd	04-05-2015 00:00	**Begindiepte monsterneming**	n.v.t.
Begindatum/tijd monsterneming		**Einddiepte monsterneming**	n.v.t.
Einddatum/tijd monsterneming			
Ontvangstdatum monster	18-05-2015 07:10		
Vrijgavedatum monster	27-05-2015 14:24		
Opmerking klant	P van Vugt		

Biochemisch zuurstofverbruik, BZV na 5 dagen — *AQUON-Leiden*

Electrochemie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
	Biochemisch zuurstofverbruik met allylthioureum	41000	mg/l	uitgedrukt in Zuurstof		2

Chemisch Zuurstofverbruik — *AQUON-Leiden*

Titrimetrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
	Chemisch zuurstofverbruik	1291800	mg/kg	uitgedrukt in Zuurstof / drooggewicht	2	2

Droge stof (Indamprest) en gloeirest — *AQUON-Leiden*

Gravimetrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	Droge stof	7.51	%	Niet van toepassing	5	2
Q	Gloeirest	25	%	t.o.v. drooggewicht	6	2

Stikstof Kjeldahl — *AQUON-Leiden*

Titrimetrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	stikstof Kjeldahl	49	g/kg	uitgedrukt in Stikstof / drooggewicht	1	2

Metalen — *AQUON-Leiden*

Inductie gekoppeld plasma - massaspectrometrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
	magnesium	3300	mg/kg	t.o.v. drooggewicht	3, 4	
Q	fosfor totaal	23	g/kg	uitgedrukt in Fosfor / drooggewicht	3, 4	
	calcium	19000	mg/kg	t.o.v. drooggewicht	3, 4	
Q	chroom	30	mg/kg	t.o.v. drooggewicht	3, 4	
Q	ijzer	13	g/kg	t.o.v. drooggewicht	3, 4	
Q	nikkel	17	mg/kg	t.o.v. drooggewicht	3, 4	
Q	koper	460	mg/kg	t.o.v. drooggewicht	3, 4	
Q	zink	640	mg/kg	t.o.v. drooggewicht	3, 4	
Q	arseen	3.0	mg/kg	t.o.v. drooggewicht	3, 4	
Q	molybdeen	12	mg/kg	t.o.v. drooggewicht	3, 4	
Q	cadmium	0.80	mg/kg	t.o.v. drooggewicht	3, 4	
Q	antimoon	2.8	mg/kg	t.o.v. drooggewicht	3, 4	
Q	kwik	0.15	mg/kg	t.o.v. drooggewicht	3, 4	
Q	lood	56	mg/kg	t.o.v. drooggewicht	3, 4	

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Nutriënten

AQUON-Leiden

Discreetanalyser

Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
chloride	5900	mg/kg	t.o.v. drooggewicht	1	2
ammonium	12000	mg/kg	uitgedrukt in Stikstof / drooggewicht	1	2

PAK

AQUON-Leiden

Hoge druk vloeistofchromatografie - UV- en fluoresc.detector

Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
naftaleen	<0.05	mg/kg	t.o.v. drooggewicht	1	
acenaftyleen	<0.05	mg/kg	t.o.v. drooggewicht	1	
acenafteen	0.20	mg/kg	t.o.v. drooggewicht	1	
fluoreen	0.69	mg/kg	t.o.v. drooggewicht	1	
fenantreen	2.6	mg/kg	t.o.v. drooggewicht	1	
antraceen	0.55	mg/kg	t.o.v. drooggewicht	1	
fluorantheen	5.9	mg/kg	t.o.v. drooggewicht	1	
pyreen	3.9	mg/kg	t.o.v. drooggewicht	1	
benzo(a)antraceen	3.3	mg/kg	t.o.v. drooggewicht	1	
chryseen	3.3	mg/kg	t.o.v. drooggewicht	1	
benzo(b)fluorantheen	3.3	mg/kg	t.o.v. drooggewicht	1	
benzo(k)fluorantheen	1.6	mg/kg	t.o.v. drooggewicht	1	
benzo(a)pyreen	2.9	mg/kg	t.o.v. drooggewicht	1	
dibenzo(a,h)antraceen	0.52	mg/kg	t.o.v. drooggewicht	1	
benzo(ghi)peryleen	1.5	mg/kg	t.o.v. drooggewicht	1	
indeno(1,2,3-cd)pyreen	2.0	mg/kg	t.o.v. drooggewicht	1	
som 10 polyaromatische koolwaterstoffen (VROM)	24	mg/kg	t.o.v. drooggewicht	1	
som 16 polyaromatische koolwaterstoffen (EPA)	32	mg/kg	t.o.v. drooggewicht	1	

Metalen

Eurofins Analytico

Inductie gekoppeld plasma - atomaire emissie spectrometrie

Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
aluminium	8900	mg/kg	t.o.v. drooggewicht		
zwavel totaal	8400	mg/kg	t.o.v. drooggewicht		

Overzicht opmerkingen

1 KS Feed IRC3

2 De conserveringstermijn voor deze analyse is overschreden, de betrouwbaarheid van het resultaat wordt hierdoor mogelijk beïnvloed.

Overzicht normen

1	Analyse	Eigen methode	
2	Analyse	Conform	NEN 6633:2006/A1:2007 nl
3	Analyse	Conform	NEN-EN-ISO 17294-2:2004 en
4	Voorbehandeling	Conform	NEN 6961:2005 nl
5	Analyse	Gelijkwaardig aan	NEN-EN 12880:2001 en
6	Analyse	Gelijkwaardig aan	NEN-EN 12879:2001 en

Legenda

De met een "Q" gemerkte parameters zijn geaccrediteerd.
De met een "S" gemerkte parameters zijn door de RvA geaccrediteerd op basis van het schema AS 2000 en AS 3000

Afkorting bij resultaten:
n.a. : niet aantoonbaar
n.g. : niet gemeten
n.t.b. : niet te bepalen

Bezoekadres AQUON locatie Tiel	De Blomboogerd 12	4003 BX	Tiel
Bezoekadres AQUON locatie Breda	Korte Huifakkerstraat 6	4815 PS	Breda
Bezoekadres AQUON locatie Leiden	Voorschoterweg 18H	2324 AB	Leiden
Postadres AQUON	Postbus 328	4000 AH	Tiel

TESTEN
RvA L 553

AQUON is geaccrediteerd volgens de door de Raad voor Accreditatie gestelde criteria voor testlaboratoria, conform NEN-EN-ISO/IEC 17025, onder nummer L553 voor gebieden zoals nader omschreven in de scope van de accreditatie. Op al onze werkzaamheden zijn de Algemene leveringsvoorwaarden van toepassing.

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Waterschap Aa en Maas Afd. Sector Zuiveren
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ANALYSERAPPORT

Datum	Code	Versie	Informatie
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Opdrachtreferentie	2015-351
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Postbus 326
4000 AH Tiel

Monsternummer	15-038790			Opm.: 1
Monsterpuntcode	302999			
Monsterpuntomschrijving	RWZI Oijen, Diversen			
Matrix	Afvalwater			
Type bemonstering	Steekmonster			
Soort onderzoek	Project			
Contramonster	Nee			
Monsternemingsdatum/tijd	05-05-2015 23:38	**Begindiepte monsterneming**	n.v.t.	
Begindatum/tijd monsterneming		**Einddiepte monsterneming**	n.v.t.	
Einddatum/tijd monsterneming				
Ontvangstdatum monster	15-05-2015 10:29			
Vrijgavedatum monster	26-05-2015 13:44			
Opmerking klant	P van Vugt			

Respiratieremming — *AQUON-Breda*

Electrochemie

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
	Toxiciteit	n.g.	%	Niet van toepassing	9	2, 3

Droge Stof (Indamprest) en gloeirest — *AQUON-Leiden*

Gravimetrie

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
	Droge stof	7000	mg/l	Niet van toepassing	14	2

PAK — *AQUON-Leiden*

Hoge druk vloeistofchromatografie - UV- en fluoresc.detector

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
Q	naftaleen	1002	ug/l	Niet van toepassing	5, 6	
Q	acenaftyleen	8.0	ug/l	Niet van toepassing	7, 8	
Q	acenafteen	10	ug/l	Niet van toepassing	5, 6	
Q	fluoreen	5.1	ug/l	Niet van toepassing	5, 6	
Q	fenantreen	1.9	ug/l	Niet van toepassing	5, 6	
Q	antraceen	0.52	ug/l	Niet van toepassing	5, 6	
Q	fluorantheen	0.49	ug/l	Niet van toepassing	5, 6	
Q	pyreen	<0.20	ug/l	Niet van toepassing	5, 6	
Q	benzo(a)antraceen	0.56	ug/l	Niet van toepassing	5, 6	
Q	chryseen	0.79	ug/l	Niet van toepassing	5, 6	
Q	benzo(b)fluorantheen	0.26	ug/l	Niet van toepassing	5, 6	
Q	benzo(k)fluorantheen	0.14	ug/l	Niet van toepassing	5, 6	
Q	benzo(a)pyreen	0.40	ug/l	Niet van toepassing	5, 6	
Q	dibenzo(a,h)antraceen	<0.10	ug/l	Niet van toepassing	5, 6	
Q	benzo(ghi)peryleen	0.16	ug/l	Niet van toepassing	5, 6	
Q	indeno(1,2,3-cd)pyreen	0.15	ug/l	Niet van toepassing	5, 6	
Q	som 10 polyaromatische koolwaterstoffen (VROM)	1007	ug/l	Niet van toepassing	5	
Q	som 16 polyaromatische koolwaterstoffen (EPA)	1031	ug/l	Niet van toepassing	5	

Biochemisch zuurstofverbruik, BZV na 5 dagen — *AQUON-Tiel*

Electrochemie

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
Q	Biochemisch zuurstofverbruik met allylthioureum	260	mg/l	uitgedrukt in Zuurstof	15	

Chemisch Zuurstofverbruik — *AQUON-Tiel*

Titrimetrie

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
Q	Chemisch zuurstofverbruik	710	mg/l	uitgedrukt in Zuurstof	2	

Kwik (Hg) — *AQUON-Tiel*

Atomaire absorptie spectrometrie - koude-damptechniek

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	kwik	<0.2	ug/l	Niet van toepassing	10	

Metalen — *AQUON-Tiel*

Inductie gekoppeld plasma - massaspectrometrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	Magnesium	6.6	mg/l	Niet van toepassing	12, 13	4, 5
Q	Aluminium	840	ug/l	Niet van toepassing	12, 13	4, 5
Q	Kalium	2500	mg/l	Niet van toepassing	12, 13	4, 5
Q	Calcium	<10	mg/l	Niet van toepassing	12, 13	4, 5, 6
Q	Chroom	1200	ug/l	Niet van toepassing	12, 13	4, 5
Q	IJzer	3.1	mg/l	Niet van toepassing	12, 13	4, 5, 7
Q	Nikkel	83000	ug/l	Niet van toepassing	12, 13	4, 5
Q	Koper	92	ug/l	Niet van toepassing	12, 13	4, 5
Q	Zink	<50	ug/l	Niet van toepassing	12, 13	4, 5, 6
Q	Arseen	<25	ug/l	Niet van toepassing	12, 13	4, 5, 6
Q	Molybdeen	1650394	ug/l	Niet van toepassing	12, 13	4, 5
Q	Cadmium	2100	ug/l	Niet van toepassing	12, 13	4, 5
Q	Antimoon	<5.0	ug/l	Niet van toepassing	12, 13	4, 5, 6
Q	Lood	<25	ug/l	Niet van toepassing	12, 13	4, 5, 6

Nutriënten — *AQUON-Tiel*

Discreetanalyser

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	chloride	170	mg/l	na filtratie (opgeloste fractie)	1	
Q	ammonium	500	mg/l	uitgedrukt in Stikstof na filtratie	1	2

Fosfor totaal — *AQUON-Tiel*

Autoanalyser

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	fosfor totaal	95	mg/l	uitgedrukt in Fosfor	3, 4	

Onopgeloste stoffen (zwevende stof) , glasvezelfilter — *AQUON-Tiel*

Gravimetrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	Zwevende stof	300	mg/l	Niet van toepassing	11	

Metalen — *Eurofins Analytico*

Inductie gekoppeld plasma - massaspectrometrie

Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
zwavel totaal	18	mg/l	Niet van toepassing		

Overzicht opmerkingen

1 Abwasser B306 Verena KS2
2 De conserveringstermijn voor deze analyse is overschreden, de betrouwbaarheid van het resultaat wordt hierdoor mogelijk beïnvloed.
3 Respiratieremming onv: 40%, 5xvv: 30%, 10xvv: 20%, 20xvv: <10%
4 Het monster is voor de betreffende analyse niet geconserveerd aangeleverd. De betrouwbaarheid van het analyseresultaat is hierdoor mogelijk beïnvloed.
5 Het monster is visueel erg vuil
6 Rapportagegrens verhoogd i.v.m. matrixstoring.
7 Resultaat is indicatief. ivm storing Cr

Overzicht normen

1	Analyse	Conform	NEN 6604:2007 nl
2	Analyse	Conform	NEN 6633:2006/A1:2007 nl
3	Analyse	Conform	NEN-EN-ISO 15681-2:2005 en
4	Voorbehandeling	Conform	NEN-EN-ISO 6878:2004 en

TESTEN RvA L 553

AQUON is geaccrediteerd volgens de door de Raad voor Accreditatie gestelde criteria voor testlaboratoria, conform NEN -EN-ISO/IEC 17025, onder nummer L553 voor gebieden zoals nader omschreven in de scope van de accreditatie. Op al onze werkzaamheden zijn de Algemene leveringsvoorwaarden van toepassing.

5	Analyse	Conform	NEN-EN-ISO 17993:2004 en
6	Voorbehandeling		NEN-EN-ISO 17993:2004 en
7	Analyse	Eigen methode	
8	Voorbehandeling	Eigen methode	
9	Analyse		NEN-EN-ISO 8192:2007 en
10	Analyse	Conform	NEN-EN 1483:2007 en
11	Analyse	Gelijkwaardig aan	NEN 6499:2010 nl
12	Analyse	Conform	NEN-EN-ISO 17294-2:2004 en
13	Voorbehandeling	Conform	NEN-EN-ISO 15587-1:2002 en
14	Analyse	Gelijkwaardig aan	NEN-EN 12880:2001 en
15	Analyse	Gelijkwaardig aan	NEN-EN 1899-1:1998 en,nl

Legenda

De met een "Q" gemerkte parameters zijn geaccrediteerd.
De met een "S" gemerkte parameters zijn door de RvA geaccrediteerd op basis van het schema AS 2000 en AS 3000

Afkorting bij resultaten:
n.a. : niet aantoonbaar
n.g. : niet gemeten
n.t.b. : niet te bepalen

Bezoekadres AQUON locatie Tiel	De Blomboogerd 12	4003 BX	Tiel
Bezoekadres AQUON locatie Breda	Korte Huifakkerstraat 6	4815 PS	Breda
Bezoekadres AQUON locatie Leiden	Voorschoterweg 18H	2324 AB	Leiden
Postadres AQUON	Postbus 328	4000 AH	Tiel

TESTEN
RvA L 553

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Instituut voor wateronderzoek en advies

Postbus 328
4000 AH Tiel

Waterschap Aa en Maas Afd. Sector Zuiveren
T.a.v. F.W. van der Molen
Pettelaarpark 70
5216 PP 's-Hertogenbosch

ANALYSERAPPORT

Datum	**Code**	**Versie**	**Informatie**
17-06-2015	R150617022	1	klantvragen@aquon.nl

Opdrachtreferentie	2015-351
Opdrachtomschrijving	2015 Voorbereidingen VERENA test KIT WsAM
Opdracht referentie klant	5.5220.054
Rapportage ontvanger	P. van Vugt

Geachte heer van der Molen,

Hierbij zend ik u de resultaten van het laboratoriumonderzoek dat op uw verzoek is uitgevoerd. Deze analyses hebben uitsluitend betrekking op de monsters, zoals die door u ter analyse zijn aangeboden.

De werkzaamheden zijn, tenzij anders aangegeven, uitgevoerd conform de PDC van AQUON. Tevens is in het "overzicht methodes AQUON" informatie over de toegepaste onderzoeksmethoden te vinden. Beide documenten zijn te vinden op de website van AQUON: http://www.aquon.nl/downloads/downloads.aspx.
Informatie over uitbestede analyses (prestatiekenmerken, accreditatie, toegepaste normen) kan worden opgevraagd bij accountmanagement van AQUON.

Het analyserapport mag slechts in zijn geheel worden gereproduceerd, tenzij vooraf schriftelijk toestemming van het laboratorium wordt verkregen.

Ik vertrouw erop uw opdracht naar tevredenheid en conform de afspraak te hebben uitgevoerd. Heeft u naar aanleiding van deze rapportage nog vragen, dan verzoek ik u contact op te nemen met het laboratorium via het bovenstaande emailadres.

Hoogachtend,

Ir. E.F.M. Nieuwenhuis
Adjunct-Directeur

TESTEN
RvA L 553

AQUON is geaccrediteerd volgens de door de Raad voor Accreditatie gestelde criteria voor testlaboratoria, conform NEN-EN-ISO/IEC 17025, onder nummer L553 voor gebieden zoals nader omschreven in de scope van de accreditatie. Op al onze werkzaamheden zijn de Algemene leveringsvoorwaarden van toepassing.

Instituut voor wateronderzoek en advies

Postbus 328
4000 AH Tiel

Monsternummer	15-042449	Opm.: 1	
Monsterpuntcode	302999		
Monsterpuntomschrijving	RWZI Oijen, Diversen		
Matrix	Afvalwater		
Type bemonstering	Steekmonster		
Soort onderzoek	Project		
Contramonster	Nee		
Monsternemingsdatum/tijd	20-05-2015 00:00	**Begindiepte monsterneming**	n.v.t.
Begindatum/tijd monsterneming		**Einddiepte monsterneming**	n.v.t.
Einddatum/tijd monsterneming			
Ontvangstdatum monster	27-05-2015 14:21		
Vrijgavedatum monster	17-06-2015 16:22		
Opmerking klant	P van Vugt		

PAK — *AQUON-Leiden*

Hoge druk vloeistofchromatografie - UV- en fluoresc.detector

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
Q	naftaleen	1619	ug/l	Niet van toepassing	6, 7	
Q	acenaftyleen	388	ug/l	Niet van toepassing	8, 9	
Q	acenafteen	259	ug/l	Niet van toepassing	6, 7	
Q	fluoreen	392	ug/l	Niet van toepassing	6, 7	
Q	fenantreen	764	ug/l	Niet van toepassing	6, 7	
Q	antraceen	227	ug/l	Niet van toepassing	6, 7	
Q	fluorantheen	641	ug/l	Niet van toepassing	6, 7	
Q	pyreen	541	ug/l	Niet van toepassing	6, 7	
Q	benzo(a)antraceen	344	ug/l	Niet van toepassing	6, 7	
Q	chryseen	348	ug/l	Niet van toepassing	6, 7	
Q	benzo(b)fluorantheen	270	ug/l	Niet van toepassing	6, 7	
Q	benzo(k)fluorantheen	129	ug/l	Niet van toepassing	6, 7	
Q	benzo(a)pyreen	250	ug/l	Niet van toepassing	6, 7	
Q	dibenzo(a,h)antraceen	34	ug/l	Niet van toepassing	6, 7	
Q	benzo(ghi)peryleen	204	ug/l	Niet van toepassing	6, 7	
Q	indeno(1,2,3-cd)pyreen	160	ug/l	Niet van toepassing	6, 7	
Q	som 10 polyaromatische koolwaterstoffen (VROM)	4687	ug/l	Niet van toepassing	6	
Q	som 16 polyaromatische koolwaterstoffen (EPA)	6570	ug/l	Niet van toepassing	6	

Biochemisch zuurstofverbruik, BZV na 5 dagen — *AQUON-Tiel*

Electrochemie

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
Q	Biochemisch zuurstofverbruik met allylthioureum	12300	mg/l	uitgedrukt in Zuurstof	2	2

Chemisch Zuurstofverbruik — *AQUON-Tiel*

Titrimetrie

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
Q	Chemisch zuurstofverbruik	37900	mg/l	uitgedrukt in Zuurstof	14	

Kwik (Hg) — *AQUON-Tiel*

Atomaire absorptie spectrometrie - koude-damptechniek

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
Q	kwik	30.6	ug/l	Niet van toepassing	13	3

Droge Stof (Indamprest) in afvalwater — *AQUON-Tiel*

Gravimetrie

	Parameteromschrijving	*Resultaat*	*Eenheid*	*Hoedanigheid*	*Normen*	*Opm*
	Droge stof	5.76	%	Niet van toepassing	12	

RvA TESTEN RvA L 553

Instituut voor wateronderzoek en advies

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4000 AH Tiel

Stikstof Kjeldahl — AQUON-Tiel

Titrimetrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	stikstof Kjeldahl	3000	mg/l	uitgedrukt in Stikstof	5	

Metalen — AQUON-Tiel

Inductie gekoppeld plasma - massaspectrometrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	Magnesium	1300	mg/l	Niet van toepassing	10, 11	
Q	Aluminium	3400620	ug/l	Niet van toepassing	10, 11	
Q	Kalium	15000	mg/l	Niet van toepassing	10, 11	
Q	Calcium	8000	mg/l	Niet van toepassing	10, 11	
Q	Chroom	22000	ug/l	Niet van toepassing	10, 11	
Q	IJzer	4700	mg/l	Niet van toepassing	10, 11	
Q	Nikkel	610000	ug/l	Niet van toepassing	10, 11	
Q	Koper	170000	ug/l	Niet van toepassing	10, 11	
Q	Zink	250000	ug/l	Niet van toepassing	10, 11	
Q	Arseen	330	ug/l	Niet van toepassing	10, 11	
Q	Molybdeen	140000	ug/l	Niet van toepassing	10, 11	
Q	Cadmium	420	ug/l	Niet van toepassing	10, 11	
Q	Antimoon	290	ug/l	Niet van toepassing	10, 11	
Q	Lood	22000	ug/l	Niet van toepassing	10, 11	

Nutriënten — AQUON-Tiel

Discreetanalyser

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	chloride	1600	mg/l	na filtratie (opgeloste fractie)	1	
Q	ammonium	1300	mg/l	uitgedrukt in Stikstof na filtratie	1	

Fosfor totaal — AQUON-Tiel

Autoanalyser

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	fosfor totaal	4400	mg/l	uitgedrukt in Fosfor	3, 4	

Metalen — Eurofins Analytico

Inductie gekoppeld plasma - massaspectrometrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
	zwavel totaal	620	mg/l	Niet van toepassing		

Overzicht opmerkingen

1 B202 Cyclone Sump Verena

2 De conserveringstermijn voor deze analyse is overschreden, de betrouwbaarheid van het resultaat wordt hierdoor mogelijk beïnvloed.

3 De overige gemeten verdunningen geven resultaten in dezelfde range, maar zijn kwalitatief minder betrouwbaar

Overzicht normen

1	Analyse	Conform	NEN 6604:2007 nl
2	Analyse	Gelijkwaardig aan	NEN-EN 1899-1:1998 en,nl
3	Analyse	Conform	NEN-EN-ISO 15681-2:2005 en
4	Voorbehandeling	Conform	NEN-EN-ISO 6878:2004 en
5	Analyse	Conform	NEN-ISO 5663:1993 en
6	Analyse	Conform	NEN-EN-ISO 17993:2004 en
7	Voorbehandeling		NEN-EN-ISO 17993:2004 en
8	Analyse	Eigen methode	
9	Voorbehandeling	Eigen methode	
10	Analyse	Conform	NEN-EN-ISO 17294-2:2004 en
11	Voorbehandeling	Conform	NEN-EN-ISO 15587-1:2002 en
12	Analyse	Gelijkwaardig aan	NEN-EN 12880:2001 en
13	Analyse	Conform	NEN-EN 1483:2007 en
14	Analyse	Conform	NEN 6633:2006/A1:2007 nl

TESTEN RvA L 553

Legenda

De met een "Q" gemerkte parameters zijn geaccrediteerd.
De met een "S" gemerkte parameters zijn door de RvA geaccrediteerd op basis van het schema AS 2000 en AS 3000

Afkorting bij resultaten:
n.a. : niet aantoonbaar
n.g. : niet gemeten
n.t.b. : niet te bepalen

Bezoekadres AQUON locatie Tiel	De Blomboogerd 12	4003 BX	Tiel
Bezoekadres AQUON locatie Breda	Korte Huifakkerstraat 6	4815 PS	Breda
Bezoekadres AQUON locatie Leiden	Voorschoterweg 18H	2324 AB	Leiden
Postadres AQUON	Postbus 328	4000 AH	Tiel

TESTEN
RvA L 553

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Instituut voor wateronderzoek en advies

Postbus 328
4000 AH Tiel

Waterschap Aa en Maas Afd. Sector Zuiveren
T.a.v. F.W. van der Molen
Pettelaarpark 70
5216 PP 's-Hertogenbosch

ANALYSERAPPORT

Datum	**Code**	**Versie**	**Informatie**
03-06-2015	R150603031	1	klantvragen@aquon.nl

Opdrachtreferentie	2015-351
Opdrachtomschrijving	2015 Voorbereidingen VERENA test KIT WsAM
Opdracht referentie klant	5.5220.054
Rapportage ontvanger	P. van Vugt

Geachte heer van der Molen,

Hierbij zend ik u de resultaten van het laboratoriumonderzoek dat op uw verzoek is uitgevoerd. Deze analyses hebben uitsluitend betrekking op de monsters, zoals die door u ter analyse zijn aangeboden.

De werkzaamheden zijn, tenzij anders aangegeven, uitgevoerd conform de PDC van AQUON. Tevens is in het "overzicht methodes AQUON" informatie over de toegepaste onderzoeksmethoden te vinden. Beide documenten zijn te vinden op de website van AQUON: http://www.aquon.nl/downloads/downloads.aspx.
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Het analyserapport mag slechts in zijn geheel worden gereproduceerd, tenzij vooraf schriftelijk toestemming van het laboratorium wordt verkregen.

Ik vertrouw erop uw opdracht naar tevredenheid en conform de afspraak te hebben uitgevoerd. Heeft u naar aanleiding van deze rapportage nog vragen, dan verzoek ik u contact op te nemen met het laboratorium via het bovenstaande emailadres.

Hoogachtend,

Ir. E.F.M. Nieuwenhuis
Adjunct-Directeur

Instituut voor wateronderzoek en advies

Postbus 328
4000 AH Tiel

Rapportnummer: R150603031 / 1

Monsternummer	15-038795		Opm.: 1
Monsterpuntcode	302999		
Monsterpuntomschrijving	RWZI Oijen, Diversen		
Matrix	Afvalwater		
Type bemonstering	Steekmonster		
Soort onderzoek	Project		
Contramonster	Nee		
Monsternemingsdatum/tijd	05-05-2015 08:30	**Begindiepte monsterneming**	n.v.t.
Begindatum/tijd monsterneming		**Einddiepte monsterneming**	n.v.t.
Einddatum/tijd monsterneming			
Ontvangstdatum monster	15-05-2015 10:32		
Vrijgavedatum monster	03-06-2015 13:06		
Opmerking klant	P van Vugt		

PAK — *AQUON-Leiden*

Hoge druk vloeistofchromatografie - UV- en fluoresc.detector

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	naftaleen	8830	ug/l	Niet van toepassing	5, 6	
Q	acenaftyleen	631	ug/l	Niet van toepassing	7, 8	
Q	acenafteen	740	ug/l	Niet van toepassing	5, 6	
Q	fluoreen	1306	ug/l	Niet van toepassing	5, 6	
Q	fenantreen	1446	ug/l	Niet van toepassing	5, 6	
Q	antraceen	441	ug/l	Niet van toepassing	5, 6	
Q	fluorantheen	278	ug/l	Niet van toepassing	5, 6	
Q	pyreen	398	ug/l	Niet van toepassing	5, 6	
Q	benzo(a)antraceen	80	ug/l	Niet van toepassing	5, 6	
Q	chryseen	151	ug/l	Niet van toepassing	5, 6	
Q	benzo(b)fluorantheen	35	ug/l	Niet van toepassing	5, 6	
Q	benzo(k)fluorantheen	19	ug/l	Niet van toepassing	5, 6	
Q	benzo(a)pyreen	65	ug/l	Niet van toepassing	5, 6	
Q	dibenzo(a,h)antraceen	6.0	ug/l	Niet van toepassing	5, 6	
Q	benzo(ghi)peryleen	26	ug/l	Niet van toepassing	5, 6	
Q	indeno(1,2,3-cd)pyreen	29	ug/l	Niet van toepassing	5, 6	
Q	som 10 polyaromatische koolwaterstoffen (VROM)	11364	ug/l	Niet van toepassing	5	
Q	som 16 polyaromatische koolwaterstoffen (EPA)	14480	ug/l	Niet van toepassing	5	

Biochemisch zuurstofverbruik, BZV na 5 dagen — *AQUON-Tiel*

Electrochemie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	Biochemisch zuurstofverbruik met allylthioureum	920	mg/l	uitgedrukt in Zuurstof	2	

Chemisch Zuurstofverbruik — *AQUON-Tiel*

Titrimetrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	Chemisch zuurstofverbruik	2510	mg/l	uitgedrukt in Zuurstof	3	

Kwik (Hg) — *AQUON-Tiel*

Atomaire absorptie spectrometrie - koude-damptechniek

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	kwik	1.7	ug/l	Niet van toepassing	9	

Droge Stof (Indamprest) in afvalwater — *AQUON-Tiel*

Gravimetrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
	Droge stof	0.03	%	Niet van toepassing	4	2

Instituut voor wateronderzoek en advies

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Metalen — *AQUON-Tiel*

Inductie gekoppeld plasma - massaspectrometrie

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	Magnesium	<1.0	mg/l	Niet van toepassing	10, 11	3, 4
Q	Aluminium	190	ug/l	Niet van toepassing	10, 11	3, 4
Q	Kalium	9.0	mg/l	Niet van toepassing	10, 11	3, 4
Q	Calcium	<2.0	mg/l	Niet van toepassing	10, 11	3, 4
Q	Chroom	41	ug/l	Niet van toepassing	10, 11	3, 4
Q	IJzer	<0.30	mg/l	Niet van toepassing	10, 11	3, 4
Q	Nikkel	5000	ug/l	Niet van toepassing	10, 11	3, 4
Q	Koper	13	ug/l	Niet van toepassing	10, 11	3, 4
Q	Zink	14	ug/l	Niet van toepassing	10, 11	3, 4
Q	Arseen	<5.0	ug/l	Niet van toepassing	10, 11	3, 4
Q	Molybdeen	1100	ug/l	Niet van toepassing	10, 11	3, 4
Q	Cadmium	1.3	ug/l	Niet van toepassing	10, 11	3, 4
Q	Antimoon	<1.0	ug/l	Niet van toepassing	10, 11	3, 4
Q	Lood	<5.0	ug/l	Niet van toepassing	10, 11	3, 4

Nutriënten — *AQUON-Tiel*

Discreetanalyser

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	chloride	25	mg/l	na filtratie (opgeloste fractie)	1	
Q	ammonium	2200	mg/l	uitgedrukt in Stikstof na filtratie	1	2

Fosfor totaal — *AQUON-Tiel*

Autoanalyser

	Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
Q	fosfor totaal	1.00	mg/l	uitgedrukt in Fosfor	12, 13	

Metalen — *Eurofins Analytico*

Inductie gekoppeld plasma - massaspectrometrie

Parameteromschrijving	Resultaat	Eenheid	Hoedanigheid	Normen	Opm
zwavel totaal	1.7	mg/l	Niet van toepassing		

Overzicht opmerkingen

1 Reahtosumpf abrug B202
2 De conserveringstermijn voor deze analyse is overschreden, de betrouwbaarheid van het resultaat wordt hierdoor mogelijk beïnvloed.
3 Het monster is voor de betreffende analyse niet geconserveerd aangeleverd. De betrouwbaarheid van het analyseresultaat is hierdoor mogelijk beïnvloed.
4 Monster is visueel erg vuil

Overzicht normen

1	Analyse	Conform	NEN 6604:2007 nl
2	Analyse	Gelijkwaardig aan	NEN-EN 1899-1:1998 en,nl
3	Analyse	Conform	NEN 6633:2006/A1:2007 nl
4	Analyse	Gelijkwaardig aan	NEN-EN 12880:2001 en
5	Analyse	Conform	NEN-EN-ISO 17993:2004 en
6	Voorbehandeling		NEN-EN-ISO 17993:2004 en
7	Analyse	Eigen methode	
8	Voorbehandeling	Eigen methode	
9	Analyse	Conform	NEN-EN 1483:2007 en
10	Analyse	Conform	NEN-EN-ISO 17294-2:2004 en
11	Voorbehandeling	Conform	NEN-EN-ISO 15587-1:2002 en
12	Analyse	Conform	NEN-EN-ISO 15681-2:2005 en
13	Voorbehandeling	Conform	NEN-EN-ISO 6878:2004 en

TESTEN
RvA L 553

Legenda

De met een "Q" gemerkte parameters zijn geaccrediteerd.
De met een "S" gemerkte parameters zijn door de RvA geaccrediteerd op basis van het schema AS 2000 en AS 3000

Afkorting bij resultaten:
n.a. : niet aantoonbaar
n.g. : niet gemeten
n.t.b. : niet te bepalen

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TESTEN
RvA L 553

AQUON is geaccrediteerd volgens de door de Raad voor Accreditatie gestelde criteria voor testlaboratoria, conform NEN -EN-ISO/IEC 17025, onder nummer L553 voor gebieden zoals nader omschreven in de scope van de accreditatie. Op al onze werkzaamheden zijn de Algemene leveringsvoorwaarden van toepassing.