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Hydrothermal carbonization of biowaste/fecal sludge

Conception and construction of a HTC prototype research unit for developing countries

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ABSTRACT

Hydrothermal carbonization (HTC) is a thermochemical process that converts biomass into a coal-like material called HTC-coal by applying high temperature to biomass in a suspension with water under saturated pressure for several hours. Whereas conversion to char via dry pyrolysis is restricted to biomass with dry water content, this process opens up the field of potential feedstock to substrates with a high moisture content such as municipal biowaste, wet agricultural residues and fecal sludge. HTC-coal is an easy to handle product with good dewatering properties and when dried, has a high calorific value. It can be used as a carbon neutral combustible, a soil conditioner, but also for a wide range of other environmental, electrochemical and catalytic applications. The interesting properties of this technology raises the question about its potential for developing countries.

To gain insights about this potential, an overview of the existing HTC-reactors already implemented in industrialized countries was conducted. Based on this outline, a HTC prototype research reactor with a simple design suitable for developing country conditions was developed, constructed and tested. The main criteria for the choice of the design were costs, material availability, complexity, durability, ease of handling and security. The assessment resulted in the decision to build a reactor made of stainless steel that is operated in a batch mode, with an internal volume of 21.8 Liters and which is heated with an electric heating mantle. The total costs for the construction, measuring equipment and certification amounts to 17'000.- CHF, of which 6000.- CHF was spent only for the certificate of the pressure vessel in conformity to the European Pressure Equipment Directives.

Two HTC tests with rice as a model substrate were carried out on the constructed HTC prototype reactor. Heat was applied during 10 hours and internal pressure, temperature and energy consumption were recorded during the process. Prescribed reactions conditions (200°C during minimum 4 hours) could be reached. The outputs were analyzed and compared with results from experiments carried out with a state-of-the-art HTC reactor of Zürich University of Applied Sciences. HTC-coal and process water with comparable characteristics than the ones coming from the reference reactor were produced. The energy consumed for the reaction was around 12 kWh on average. HTC-coal with a calorific value of 27 MJ/kg could be produced. The reactor could be operated in a safe and convenient way and can be used to conduct further experiments with waste feedstock (e.g. kitchen- and market waste, fecal sludge). The study concludes with recommendations regarding the use and safety of the reactor, regarding possible ameliorations as well as regarding the implementation of the technology in developing countries. Finally a SWOT analysis is done to critically assess the underlying concept, design, construction and operational experiences of the prototype.

CONTENTS

TABLE OF FIGURES

LIST OF TABLES

1. INTRODUCTION

1.1 BACKGROUND AND PROBLEM STATEMENT

Hydrothermal carbonization (HTC) is a thermo-chemical process used to convert biomass into a coal-like material with a higher carbon content. It is realized by applying high temperature (180-220°C) to biomass in presence of water under saturated pressure during several hours. Due to the need for efficient biomass technologies and due to its particularities and advantages over other conversion processes, HTC has recently regained considerable interest. HTC is seen as a promising technology to transform wet biomass waste streams into a coal-like product that can be used as a renewable combustible or a soil conditioner but also for a wide range of other environmental, electrochemical and catalytic applications. Another advantage is that the substrate can be hygienized during the HTC process. Therefore it can be seen as a potential technology to treat problematic biomass streams like industrial waste, biowaste or sewage sludge (Glasner, et al., 2011). In fact, the first pilot plants have already been put in operation for this purpose in Europe, and especially in Germany. However the implementation of the technology seems to be restricted to high cost and high-tech solutions suitable for industrialized countries.

The interesting properties of this technology raise the question about its potential for developing countries as well. Low and middle income countries face serious challenges with the management of fecal sludge and municipal solid waste where inadequate collection, recycling or treatment and uncontrolled disposal of waste lead to severe health and environmental problems [13]. An appropriate experience with the design and construction of a HTC reactor is needed to assess the suitability of the technology for developing countries.

1.2 RESEARCH OBJECTIVE

The overall objective is to design, construct and test a HTC prototype reactor for research purposes that can be used to assess the suitability of the technology for decentralized organic waste and/or sewage sludge treatment in developing countries. The prototype reactor needs to be designed in accordance with technical requirements to ensure proper functionality and safe operation, and adapted to conditions in developing countries.

1.3 METHODOLOGY

1.3.1 Overview of HTC

In a first step, the underlying science of HTC process and the different HTC technologies are studied in detail. The objective is to know how the HTC process works and what are its conditions and requirements; what are the input and output characteristics and what are the different designs for HTC reactors. This is carried out with literature review, by contacting specialists (researchers, engineers), conducting interviews and visits to existing HTC plants.

1.3.2 Design and construction of the HTC prototype reactor

Based on this knowledge, the design of a HTC prototype reactor for experimental purposes for developing countries is carried out. The necessary requirements that have to be observed to ensure proper functionality and safe operation of the reactor are first investigated as well as the requirements to ensure appropriateness for application in developing countries. Possible design options are then identified. The option that suits best the requirements is then selected, designed in details and constructed.

1.3.3 Test and assessment of the prototype reactor

After building the HTC reactor, the functionality of the system is tested. Hydrothermal carbonization of an organic model substrate is carried out under standard carbonization conditions. The output products are analyzed and compared with the results from experiments with a different state-of-the-art HTC reactor using the same substrate and carbonization conditions. Based on these results, a SWOT analysis is done to critically assess the underlying concept, design, construction and operational experiences of the prototype.

2. OVERVIEW OF HTC PROCESS

2.1 INTRODUCTION

Coal is formed by the decomposition of organic plant matter. In nature this gradual transformation takes place in the course of millions of years. Plants and trees covered by stagnating water go first through a decomposition phase (for example in peat bogs) and after long periods of time, sink in deeper layers (Bergius, 1913). As it sinks, the pressure and surrounding temperature increase and the organic matter gradually go through a thermochemical transformation. In this process, the hydrogen and oxygen contents of the material decrease while H_2O and CO_2 are released from the molecular structure (Krause, 2010). This leads to an increase of the carbon content and to the formation of different kinds of coals depending on the degree of transformation. The higher the degree of transformation is, the higher the carbon content. Example of coals are, from lowest to highest carbon content: lignite, sub-bituminous coal, bituminous coal, anthracite (Taylor, et al., 2009).

The conversion of biomass into products with higher carbon contents can take place by means of different thermochemical processes. Pyrolysis is for example a process which occurs under high temperature and in the absence of oxygen, and leads to the formation of charcoal (Libra, et al., 2011). When pyrolysis is carried out in the presence of sub-critical liquid water, at high temperatures and pressures, the process is called wet pyrolysis or hydrothermal carbonization (HTC) (Ramke, et al., 2009).

HTC was first used and described by the German chemist and Nobel prize winner Friedrich Bergius in the year 1913 as a means to simulate the natural coalification of organic matter in the laboratory (Funke, et al., 2010). The long time periods known from the process in nature were replaced in the laboratory with a high process temperature, thereby accelerating the kinetics of the chemical reactions.

This process was then brought to light again and investigated in more details at the Max Planck Institute of Colloids and Interfaces in Golm/Potsdam in 2006 by Prof. Markus Antonietti (Ramke, et al., 2009). Since then, it has attracted the interest of many researchers for numerous reasons.

2.1.1 Advantages of HTC

Compared to biological treatment methods (like anaerobic digestion, or alcoholic fermentation), carbonization of biomass has various advantages. First the reaction only takes hours compared to days or months needed for biological processes. Furthermore, the high process temperatures eliminate pathogens and inactivate other potential contaminants like pharmaceuticals making the outputs products sterile and hygienic (Libra, et al., 2011).

Compared to dry pyrolysis, which requires biomass with low water content (typically wood or crop residues), the main advantage of HTC is that the feedstock doesn't need to be dried before or during the process, allowing the conversion of organic matter with high water content. HTC can thus be applied to a wide range of biogenic substrates from feces to municipal biowaste and AD digestate. This process is thus particularly suitable for wet biomass as the energy intensive drying can be avoided. Furthermore, HTC requires lower process temperature (180-250°C compared to 400°C for dry pyrolysis, see table 2).

The resulting suspension (water-carbon mixture) from HTC process is easily dewatered. Comparisons between dewatering curves from wet biomass before and after HTC indicate that dewatering properties are actually much better after HTC. This makes it an interesting application for the energetic use of organic waste with high moisture content for example in wastewater treatment plants, where the dewatering of fecal sludge for incineration requires a lot of energy.

In addition, HTC is seen as an efficient process for carbon sequestration to mitigate climate change. Compared to other conversion processes that transform carbohydrates into products with higher carbon contents or other burnable fuels, HTC is in fact the most efficient. When biomass is composted, anaerobically digested or fermented, some of the original carbon in the substrate is converted into CO_2 and lost to the atmosphere. With HTC however, most of the original carbon present in the substrate stays bound to the final coal product (Titirici, et al., 2007).

2.1.2 Feedstock

HTC has a high flexibility on the choice of feedstock. In principle, any kind of biomass can be hydrothermally carbonized (Funke, et al., 2010). Substrates like stabilized and non-stabilized sewage sludge, animal manure, municipal solid waste, agriculture residues and algae are often reported in the literature to be used as input materials (Libra, et al., 2011). Conclusive experiments have also been carried out using plastic [4] or unsorted municipal solid waste (Berge, et al., 2011). HTC is typically carried out using a feedstock with water content of 75-90% or higher. Under 40%, it is unlikely that HTC has any energetic advantage (in terms of input of external heat) over dry pyrolysis (Libra, et al., 2011).

The feedstock is usually shredded such that the mixture with water can be pumped or stirred easily. Grinding of the feed may be an advantage because hydrolysis, the first step of the HTC process, is diffusion controlled and seems to be a rate determining step. Therefore the smaller the feed, the faster the reaction. However, since this implies a higher energy demand and higher investment costs it is unclear whether this leads to a significant advantage (Funke, et al., 2010).

2.2 HTC TRANSFORMATION PROCESS

During hydrothermal carbonization both the oxygen content (characterized by O/C ratio) and the hydrogen content (characterized by H/C ratio) of the feedstock are reduced which results in an augmentation of the C content. For the hydrothermal carbonization to take place, the biomass needs to be submerged in liquid water and heated, while leaving the water in subcritical condition (below 374.15°C/221.2 bars). Above this, the supercritical state of water is reached and the product is mainly gaseous (hydrothermal gasification) (Libra, et al., 2011). First reactions have been observed above 100°C, however according to Funke and Ziegler (2010) it is unlikely that practical implementations of hydrothermal carbonization happen outside the temperature range of 180-250°C. Typical residence times vary from an hour to several days (Funke, et al., 2010).

2.2.1 Reaction mechanisms

The reaction mechanisms that transform biomass into HTC-coal are similar to those in dry pyrolysis and include hydrolysis, dehydration, decarboxylation, condensation polymerization and aromatization (Libra, et al., 2011). The detailed nature of these reaction pathways is not well known and depends on the type of feed (Funke, et al., 2010). Bergius (1913) described the hydrothermal carbonization of cellulose with the following formula :

$$\begin{aligned} &(C_6H_{12}O_5)_4 \rightarrow C_{21}H_{16}O_2 + 3CO_2 + 12\,H_2O \\ &\Delta H_R = -297.9\ \text{kJ/mol} \end{aligned} \tag{1}$$

The presence of water in subcritical conditions at elevated temperatures enhances the solvent properties of water and facilitates **hydrolysis** of organic compounds (Funke, 2012). During hydrolytic reactions, the presence of water leads to the cleavage of chemical bonds of the biomacromolecules. Hydrolysis has lower activation energy than most of the reaction taking place during dry pyrolysis which leads to lower decomposition temperatures (Libra, et al., 2011). Under hydrothermal conditions, cellulose is significantly hydrolyzed above approximately 200°C (Funke, et al., 2010), hemi-cellulose between 180-200°C, and lignin is decomposed between 180-220°C (Libra, et al., 2011).

During chemical **dehydration**, the biomass is carbonized significantly by lowering the H/C and O/C ratios. For example the dehydration of glucose was formulated by Titirici et al. (2007) as follows (formula also used to describe in a simplified way the whole transformation process of carbohydrates):

$$\begin{aligned} &C_6H_{12}O_6 \rightarrow C_6H_4O_2 + 4\,H_2O \\ &\Delta H_R = -1040\ \text{kJ/mol} \end{aligned} \tag{2}$$

or also commonly found (mentioned for example by Ramke et al. 2009, Röthlein 2006, Buttmann 2011):

$$C_6H_{12}O_6 \rightarrow C_6H_2O + 5\,H_2O$$
$$\Delta H_R = -950\ \text{kJ/mol} \quad (3)$$

During **decarboxylation**, carboxyl (-COOH) and carbonyl (-C=O) groups are degraded, yielding respectively CO_2 and CO. This process happens rapidly at a temperature above 150°C. The elimination of hydroxyl (-OH) and carboxyl groups lead to the creation of unsaturated fragments of biomacromolecules. Some of these fragments are highly reactive and join together mainly by **condensation polymerization,** process in which two molecules join together leading to the loss of a small molecule (often H_2O)**.** Aromatic structures which result from the **aromatization** of polymers are very stable under hydrothermal conditions and are therefore considered as the building blocks of the HTC-coal (Funke, et al., 2010).

2.2.2 Energetic aspects of the reaction

Hydrothermal carbonization is mostly described in literature as an exothermic process, during which part of the chemical energy contained in the feedstock is released in form of heat, which is reflected in the negatives signs of the enthalpy of reaction ΔH_R of equations (1),(2) and (3). For this energy to be released and utilized, the activation energy of the reaction has to be overcome. According to Titirici (2007), once activated, HTC is a spontaneaous process liberating up to a third of the combustion energy stored in the carbohyrates through dehydration (equations (2) and (3) above).

A study by Ramke et al. (2009) compared the energy content of the input and output material using the gross calorific values[1]. It was found that 60% – 90% of the gross calorific value of the input material is available in the resulting HTC-coal. The rest being released as heat during the process (exothermy) or chemically bonded in carbon compounds dissolved in the liquid phase. The amount and proportion of heat released depends on the feedstock and on the process conditions.

However, the heat released by the exothermal reaction doesn't compensate the heat losses during the process, as indicated in a paper by Buttmann (2011) in which an energy balance of HTC of partly stabilized sewage sludge was carried out. Therefore an external heat has to be provided in order to sustain the reaction. Glasner (2011) also reports that up to date the HTC process hasn't been maintained without external energy supply.

In order to optimize the energy balance of a HTC system, an efficient heat recovery system is necessary. Reduction of heat losses can be achieved by recirculating the hot process water, which at the same time increases the residence time of the organic compounds dissolved in the water (Funke, 2012).

[1] The term "calorific value" will be used further in this report and refers to the higher heating value

2.3 INFLUENCE OF PROCESS PARAMETERS

2.3.1 Temperature

Temperature seems to be the process parameter that has the biggest influence on products characteristics. High temperatures lead to higher reaction rates, and have a decisive influence on the number of biomass compounds that can be hydrolyzed. Substantial hydrolysis starts at a temperature of about 180°C. The *reaction severity "f"* has been defined to model the influence of temperature and residence time on the products. Both high temperatures and longer residence times increase reaction severity. The higher the reaction severity is, the higher the carbon content of the HTC-coal produced (Funke, et al., 2010).

$$f = 50 \cdot t^{0.2} \cdot e^{-\frac{3500}{T}} \tag{4}$$

This suggests that if a HTC reaction is carried out with a lower temperature, a similar HTC-coal can be produced by adjusting the residence time.

2.3.2 Residence time

Exact residence time cannot be given since reaction rates remain largely unknown but typical residence times vary between 1 and 72h. Experiments with short residence time (less than an hour) have been carried out and also resulted in a significant increase of heating value of the HTC-coal produced (Funke, et al., 2010). However a longer residence time leads to higher reaction severity and reduces the amount of organic losses in the wastewater. An economical way of increasing the residence time would be by recirculation of the process water.

2.3.3 Pressure

Pressure should be such that the water remains in the liquid phase. In a close compartment, the rise in pressure is a result of rise in temperature and if the temperature rises above 100°C then the resulting vapor pressure in the compartment is the saturated pressure of water, which means that further evaporation of water will lead to condensation of the same amount of water vapor (equilibrium). For example, in a pressure vessel with temperature from 180-220°C, the resulting water vapor pressure (saturated vapor pressure) ranges between 9-22 bars (Ramke, et al., 2009).

If a pressure vessel heated above 100°C contains biomass as well, the resulting pressure will be higher than the saturated vapor pressure due to the formation of gases. For example, during HTC of biomass, the pressure in a reactor heated at 185°C can reach 22-24 bars (Glasner, et al., 2011). Using high temperatures to increase the reaction severity can result in high pressures that may imply high investment cost for pressure equipment (Funke, et al., 2010).

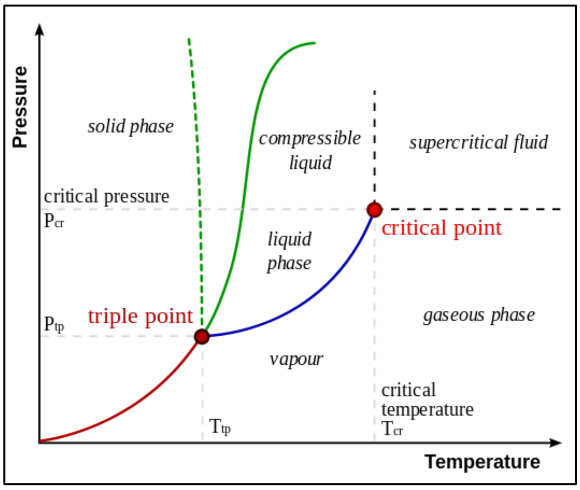


Figure 1: T-P phase diagram of water
Source: http://en.wikipedia.org/wiki/Phase_diagram

Table 1: Saturated steam table		
Saturation Temperature	Pressure	Density of Water
°C	bar gauge	kg/m³
160	5.16	907.5
170	6.90	897.5
180	9.01	887.1
190	11.53	876.1
200	14.52	864.7
210	18.05	852.8
220	22.17	840.3
230	26.94	827.3
240	32.43	813.5
250	38.72	799.1

Source: http://www.spiraxsarco.com/resources/steam-tables/saturated-water.asp

2.3.4 pH

The addition of acids has an influence on the kinetics of the reaction as well as on the reaction conditions. Weakly acid conditions tend to increase the overall rate of the HTC and increase the carbon yield as well as carbon content of the HTC-coal. Neutral to weakly acidic conditions seem to be necessary for simulating natural coalification. However a too low pH value can have a inhibiting effect on the HTC reactions (Krause, 2010).

2.3.5 Solid load

The solid load is the ratio of biomass to water. A high solid load can result in a lower overall residence time, by increasing the rate at which the concentration of monomers is raising, which allows the polymerization to start earlier (Funke, et al., 2010). In order to maximize the coal production in a reactor, the solid load should be as high as possible (Krause, 2010) but in a way that the input biomass is completely covered with water and that the mixture is able to be pumped or stirred if device such as pumps or stirrer are used.

2.4 Products of HTC

Products from HTC are in a solid, liquid and gaseous state. Typical product yields in the different phases after HTC are shown in the following table. For comparison, the product yield after dry pyrolysis is also represented.

Table 2: Comparison of product distributions (adapted from Libra et al. 2011)

	HTC	Dry pyrolysis: slow	Dry pyrolysis: fast
Reactions conditions	180-250°C, 1-12h	~400°C; h-week	~500°C; ~1s
Solid [% weight]	50-80	35	12
Liquid [% weight]	5-20	30	75
Gas [% weight]	2-5	35	13

This distribution depends strongly on the type of feedstock used and the reaction conditions (temperature, residence time, TS content). Table 3 shows the distribution of carbon (C) in all three phases for different substrates after HTC.

Table 3: Distribution of the carbon fraction in in the HTC product phases (adapted from Ramke et al. 2009)

Substrate	C in solid [%]	C in liquid [%]	C in gas [%]
Organic waste	74.9	19.0	6.1
Green cutting	75.3	19.7	5.0
Biogas slurry	72.2	22.1	5.7
Straw	75.4	19.7	4.9
Chipped wood	82.9	14.1	3.0

After the process, typically, around 14-19% of the organic carbon originally present in the substrate remains in the liquid part in form of TOC and only 3 to 6% of the C is transformed in form of gas. The remaining 72 - 83% of the C from the original biomass is thus bound in the solid part.

2.4.1 Solids

After HTC, the solid part, called HTC-coal or hydrochar, is separated from the liquid (usually by filtration). HTC-coal has a structure resembling natural coal (approaching lignite or even sub-bituminous coal depending of the reaction severity) (Funke, et al., 2010).

2.4.1.1 Characteristics of HTC-coal

The main characteristic of HTC coal is that it has higher C content and lower H/C and O/C ratios than the initial substrate. This results from the dehydration and decarboxylation processes during HTC. The following table shows examples of the mass yield and composition of HTC-coal from different substrates.

Table 4: Examples of solid yields and elementary compositions of HTC-coals from different substrates (source: Funke 2012)

	Solid yield [% dry substance]	C [% dry ash-free]	H [% dry ash-free]	O [% dry ash-free]	Reference
Cellulose		44.4	6.2	49.4	Schumacher et al 1960
HTC: 225°C, 3h	63	51.9	5.6	42.5	
Biowaste		54.6	7.5	37.9	Ramke et al. 2010
HTC: 230°C, 4.5h	57	70.5	6.9	22.6	
Food waste		45.7	6.2	43.9	Berge et al. 2010
HTC: 250°C, 20h	46	75.2	6.4	11.1	
Digestate (biogas slurry)		51.8	6.8	37.9	Mumme et al. 2010
HTC: 230°C, 6h	51	72.6	7.2	15.6	
Wood		50.3	6.0	43.3	Yan et al. 2010
HTC: 230°C, 5 min.	75	56.1	5.9	37.9	

A graphic representation as in the coalification diagram (or van Krevelen diagram) allows visualizing the hydrothermal carbonization process. In the diagram the hydrogen/carbon molar ratio is plotted against the oxygen/carbon molar ratio. During the process, both ratios are decreased and a dot representing the substrate at its initial state moves towards the downwards-left direction during the carbonization process. The degree of carbonization can be visualized by the length of the vector that binds the two dots representing the input and the output material. The following figure shows a van Krevelen diagram with the representation of input and output values for different substrates.

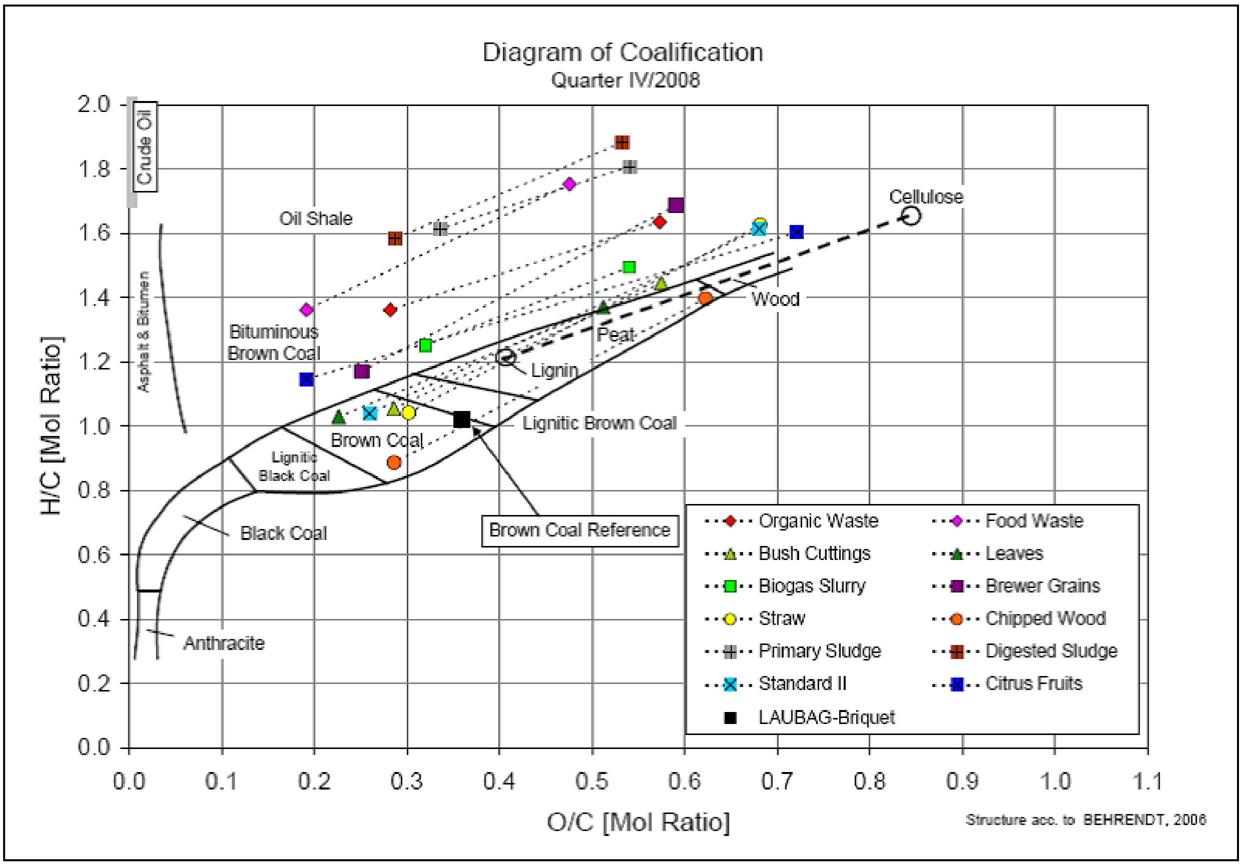


Figure 2:Van Krevelen diagram (source: Ramke et al. 2009)

With respect to calorific value and C-content, HTC-coal can be classified as being similar to brown coal (Ramke, et al., 2009). This value depends on the type of feedstock and process parameters used during the reaction. The following figure shows the calorific values (“Brennwert” in diagram) for different substrates after hydrothermal carbonization. Calorific values of brown coal (“Braunkohle”) and bituminous coal (“Steinkohle”) are represented on the right for comparison (④).

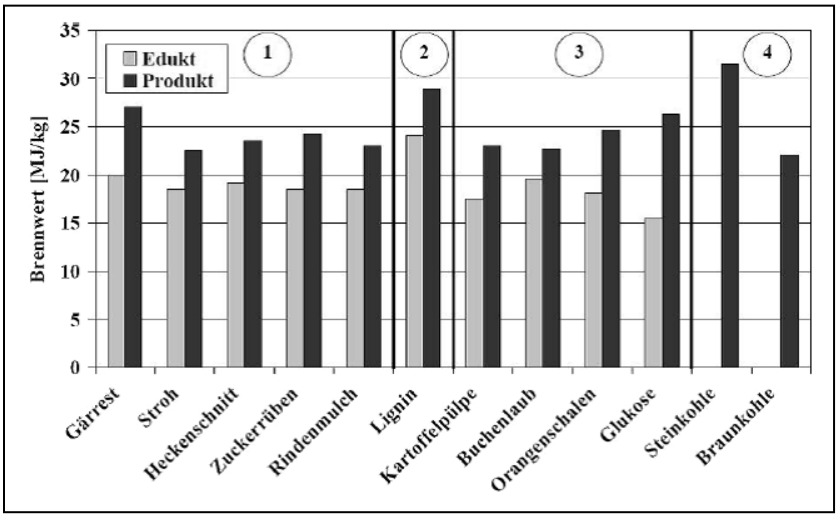


Figure 3: Comparison of calorific values of different substrates before and after HTC (source: Glasner et al. 2011)

In principle, the concentration of C, H, O and N determines the calorific value. A study by Ramke et al. (2009) with HTC-coals from different substrates shows a linear correlation between the gross calorific value and the carbon content.

One interesting characteristic of HTC-coal is that the elimination of hydroxyl and carboxyl groups during the HTC process leads to a product with a lower hydrophilicity than the initial substrate (Funke, et al., 2010), making the dewatering process of the HTC-coal easier as compared to the original biomass before the process.

2.4.1.2 Post-processing

Tests by Ramke et al. (2010) show that HTC-coal can easily be separated from the water. By using a press, wet HTC-coal from different substrates was put under constant pressure of 15 bars. The total volume of the discharge water was measured and related to the original mass of water. The corresponding TS content was calculated over time. The following figure shows the progress of the TS content of the HTC-coal from sewage sludge over time during the dewatering process compared to that of the non-carbonized sewage sludge.

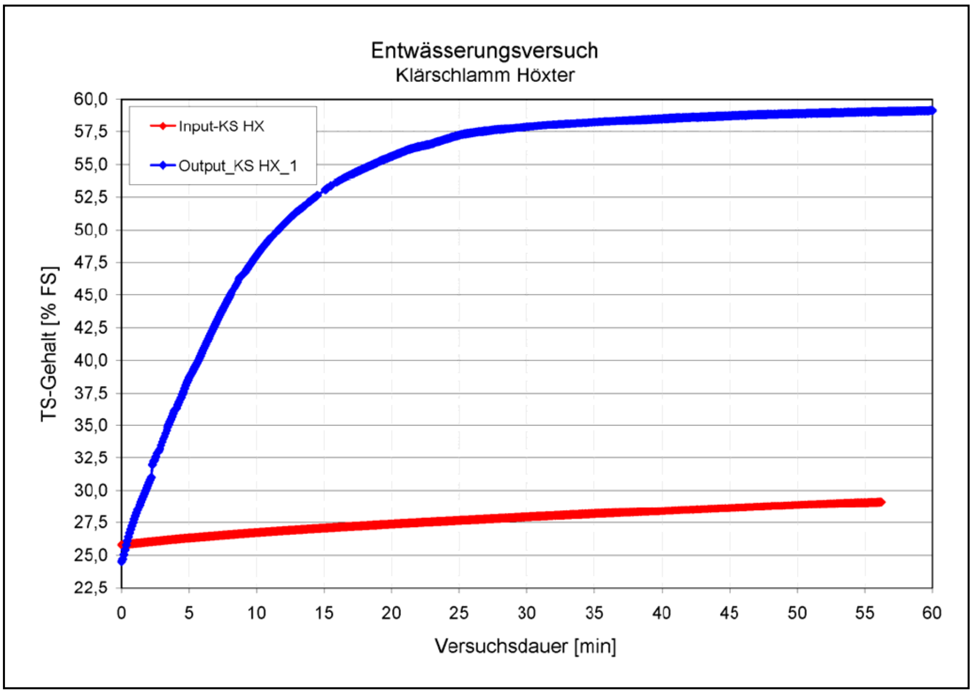


Figure 4: Dewatering diagram of sewage sludge before and after HTC (source: Ramke et al. 2010)

This result suggests that the mechanical dewatering properties of HTC-coal are significantly better than that of the original biomass and the same properties can be expected from HTC-coal from other substrates with high moisture content.

In a study by Buttmann (2011), dewatering experiments using a filter press were carried out with cold coal-suspension from HTC of sewage sludge. The resulting HTC-coal was then pelletized. The following table compares the properties of the HTC-coal pellets (8mm) with that of the sewage sludge before HTC.

Table 5: Specific energy content per volume unit for sewage sludge before and after HTC (source: Buttmann **2011)**

	Sewage sludge before HTC	Pellets of HTC-coal (8mm)
Water content [%]	80	10
Higher heating value (dry basis) [MJ/kg]	14.4	16.5
Mass density [kg/L]	1.1	0.81
Specific energetic content [MJ/L]	3.17	12.03

These results show that the specific energy content of sewage sludge can be increased by a factor 4 approximately. This can be achieved thanks to a reduction of the water content and an increase of the heating value.

2.4.2 Liquids

2.4.2.1 Characteristics of process water

The process water is the liquid that remains after the filtration of the coal suspension produced through HTC of biomass. It usually contains a high load of organic and inorganic compounds (Funke, et al., 2010), a part of the nitrogen, phosphorus as well as mineral components of the original biomass (Glasner, et al., 2011). The following table compares the values from the analysis of the process water that was made by Ramke et al. (2010) for various substrates and by Escala et al. (2012) with sewage sludge.

Table 6: Composition of the process water resulting from HTC

	Values from Escala et al. (2012)	Values from Ramke et al. (2010)
pH	5.0 – 7.0	3.7 – 7.2
Phenole [mg/L]	292 – 666	
NH_4-N [mg/L]	1053 – 2187	3.4 – 4.1
NO_3-N [mg/L]	45 – 178	2.9 – 36
NO_2-N [mg/L]	0.22 – 1.35	
Total Nitrogen [mg/L]	2263 – 4720	
PO_4-P [mg/L]	4.8 – 148.7	0.2 – 550
Total Phosphorus [mg/L]	14.3 – 159.6	
COD [mg/L]	31 467 – 53 000	14 350 – 69 610
BOD [mg/L]		10 000 – 42 000
TOC [mg/L]		9 045 – 27 840

The process water is in most of the cases acidic because of the acidic byproducts formed during the reaction and has a high COD level. The TOC represents the dissolved carbon that couldn't stay bound to the HTC-coal.

Studies by Ramke et al. (2010), showed that nutrients as well as metals in the process water don't play a significant role. It is not yet clarified to which extent possible harmful substances as well as heavy metals are present in the process water (Glasner, et al., 2011).

2.4.2.2 Post-processing

Test by Ramke et al. (2009) confirmed the good biodegradability of the dissolved organic components in the liquid phase. The efficiency of COD degradation with aerobic treatment steps reached 85%. Other experiments by Blöhse (2012) showed that the organic content of the HTC process water can be in most cases anaerobically digested, increasing the proportion of carbon that can be used energetically (carbon efficiency) by 5%.

2.4.3 Gases

The gas formed during HTC consists mainly of CO_2 due to the process of decarboxylation. The CO_2 concentration in the gas lays between 70 − 90% depending on substrate and severity of reaction (Ramke, et al., 2009). Other gases present in minor fraction are CO, CH_4 and H_2.

2.5 Use of HTC-coal

2.5.1 Renewable energy carrier

One of the main applications of HTC-coal is to use it as a combustible. As the CO_2 emitted during the combustion is balanced by the CO_2 captured during the biomass growth, it is considered a carbon neutral energy source. As previously mentioned, the specific energetic content of HTC-coal (resulting from a higher TS and a higher calorific value) can be significantly increased compared to the original feedstock. There are various options in which HTC-coal can be used as a combustible: combustion plants, combined heat and power plants, cement and steel factories, mono-combustion plants for sewage sludge, gasification [10]. In developing countries, HTC-coal can be used as cooking fuel in improved cooking stoves replacing firewood or charcoal derived from wood which could in consequence have a positive impact on deforestation.

A promising application of HTC is for the treatment of sewage sludge from a waste water treatment plant (WWTP). A study by Escala et al. (2012) compared a scenario where sewage sludge is dried and incinerated in a combustion plant, with a scenario where the sewage sludge is hydrothermally carbonized, mechanically dewatered and then incinerated. The study concludes that around 10% of energy and up to 75% of the cost for waste management can be saved per year with the application of HTC. Furthermore, it could improve the CO_2-balance by 95%.

In future, mono-combustion of sewage sludge (i.e combustion of dried sewage sludge only, without other type of wastes) is planned to be implemented in Switzerland to facilitate the recovery of phosphorus from the ash [11]. Thus, solutions which can provide a substrate with high specific energetic content like HTC will probably have a big role to play.

2.5.2 Soil amendment

Another application of HTC-coal is its use as water- and ion binding component to improve soil quality (Libra, et al., 2011). The use of charcoal as a soil conditioner (biochar) is reported to have positive effects on soil fertility (Glaser, et al., 2001 and 2002). Charcoal has a high surface area due to its porous structure which improves the water retention when applied to the soil. Furthermore, it improves the nutrient retention capacity of the soil, which increases the nutrient supply for the plant and decreases the nutrient losses by leaching. Two processes are assumed to be responsible for this. First nutrients are trapped in the fine pores of the carbonized material and secondly, slow biological oxidation produces carboxylic groups on the edges of the aromatic backbone of the charcoal which increases its nutrient holding capacity (Glaser, et al., 2001 and 2002). It is likely that HTC-coal will have similar effects on the soils due to its similar physical and chemical properties. However HTC-coal is produced at lower temperatures and may not have the same large internal surfaces as biochar (Libra, et al., 2011).

Researchers often refer to Terra Preta soils to illustrate the enhancing effect of biochar in soils. Terra Preta (black soil in Portuguese) is a dark colored soil found in Brazilian Amazon Basin most likely created by pre-Columbian Indians. It is characterized by higher levels of soil organic matter, higher moisture holding capacity, higher levels of nutrient holding capacity and nutrients such as nitrogen, phosphorus, potassium, calcium than in surrounding soils. A key factor for this enhanced fertility seems to be the high contents of anthropogenic charcoal found in the soil originating from residues of incomplete combustion mainly from cooking fires (Glaser, et al., 2001).

2.5.3 Carbon sequestration

Biomass is an efficient carbon converter, binding atmospheric CO_2 through photosynthesis. However it is only a short term carbon sink, as microbial decomposition of biomass liberates the amount of CO_2 that was bound in the plant material (Titirici, et al., 2007). It is assumed that C entering the soil as charcoal is very stable and can persist over centuries due to its chemical stability caused by the aromatic structure. Additionally this complex structure makes it resistant to microbial degradation (Glaser, et al., 2002). This long term stability has been shown in Terra Preta soils, which are on average 500-2000 years old. Through hydrothermal carbonization of biomass, the carbon can be fixed into the coal product with a very high efficiency. Therefore C entering the soil as HTC-coal can act as a significant carbon sink for atmospheric CO_2.

2.5.4 Activated carbon adsorbents

One important application field for chars is adsorption, especially for water purification. Chars can be activated to increase their pore size and surface area. Thanks to their increased sorption capacity, activated carbons can be used to adsorb a large variety of contaminants from water. Chars can be activated with two methods: physical and chemical activation. Physical activation is carried out with activating agents such as CO_2 or steam. Chemical activation is carried out by mixing the chars with chemical activating agents (such as potassium salts, sodium hydroxide, magnesium chloride,…) and heating the mixture at various temperatures in an inert environment. Sorbent materials for the removal of heavy metals have also been successfully produced using HTC without the need of an activating step.

2.5.5 Other applications

Recent research showed that hydrothermal carbonization can be used for the production of nanostructured carbonaceous material from biomass by choosing the right type of feedstock and through the addition of certain compounds. The properties of these spherically shaped nanoparticles can be interesting for various applications such as production of catalysts, carbon fixation or the production of adsorbents (Hu, et al., 2008 and Titirici, et al., 2007). Furthermore, coal particles produced with HTC show promising potential for other important applications such as hydrogen storage, electrochemical energy storage with lithium-ion batteries or supercapacitors or as feed material for fuel cells (Libra, et al., 2011).

2.6 HTC REACTORS: STATE OF THE ART

This section outlines different existing methods used for the hydrothermal carbonization of biomass. Ten different systems where reviewed with the aim to get an overview of the various technologies, as well as the degree of complexity and the various scales at which HTC reactors can exist.

2.6.1 Grenolmatik ZHAW

The experimental HTC reactor is located in ZHAW Wädenswil (Zurich University of Applied Sciences) and used for research purposes. It is operated by a PhD student and a research associate.

Figure 5: Picture of the Grenolmatik 25 at ZHAW (photo Robbiani)

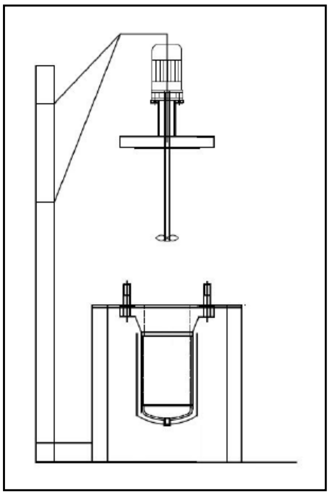


Figure 6: Schema of the Grenolmatik 25

Table 7: Characteristics of the reactor 1	
Name of the system	Grenolmatik 25
Manufacturing company	Grenol GmbH, Germany
Start of operation	November 2010
Feed mode	Batch
Volume	25 Liters
Heating system	Oil mantle. The thermal oil is heated with two 8kW-resistors
Pressure range	< 40 bars
Temperature range	20-220°C
Measurement system	2 internal pressure sensors and an internal Thermometer. Values recorded automatically
Components	- Double walled ressure chamber (stainless steel), supported by a table frame - Stirrer and manually activated crane - Heating system controlling oil temperature (electronic control) - The heating system possesses a connection for cooling with water
Security system	Overpressure valve + over pressure interrupter. In case of emergency an interrupter switches off the whole system: the safety valve opens, the stirrer stops rotating, and the heating system switches on cooling mode.
Cost	100'000 CHF
Contact	Gabriel Gerner [gega@zhaw.ch]
Address	ZHAW Zürcher Hochschule für Angewandte Wissenschaften IUNR Institut für Umwelt und Natürliche Ressourcen Grüntal, Postfach CH-8820 Wädenswil Phone: +41 58 934 54 56 Homepage: www.iunr.zhaw.ch/erneuerbareenergien
Remarks	The reactor consists of a cylindrical pressure chamber that can be filled with biomass and water. The feedstock is fed in a detachable stainless steel container to facilitate the handling of hot products. The pressure chamber is sealed with large screws, and tightened with a long torque wrench. No sampling possible during reaction.

2.6.2 Autoclave ZHAW

The autoclave was used by the ZHAW research group before the Grenolmatik 25 was bought. This is the smallest system on the list. It is useful for testing the HTC-feasibility of different substrates.

Figure 7: Picture of the autoclave at ZHAW (photo Robbiani)

Table 8: Characteristics of the reactor 2	
Name of the system	High-pressure laboratory autoclave Model II
Manufacturing company	Carl Roth GmbH
Start of operation	-
Feed mode	Batch
Volume	250 mL
Heating system	Electrical mantle
Pressure range	up to 200 bar
Temperature range	-20 - 300°C
Measurement system	Temperature sensor, pressure gauge
Components	Pressure vessel (stainless steel), heating mantle, magnetic stirrer
Security system	Bursting disc to avoid overpressure
Cost	Autoclave: CHF 1148.60 Heating mantle + stirring system: CHF 3120.-
Contact	Gabriel Gerner [gega@zhaw.ch]
Address	ZHAW Zürcher Hochschule für Angewandte Wissenschaften IUNR Institut für Umwelt und Natürliche Ressourcen Grüntal, Postfach CH-8820 Wädenswil Phone: +41 58 934 54 56 Homepage: www.iunr.zhaw.ch/erneuerbareenergien
Remarks	Simple cylindrical pressure container, sealed with large screws. The stirring for some substrates is difficult with such a small autoclave. This is why a bigger reactor was bought.

2.6.3 Diving bottles

At the beginning of the research about HTC at ZHAW, an experiment was carried out by Christoph Koller where biomass was fed in diving bottles and heated on an open fire. This is an example of a very simple and low cost option, but also a very inefficient and dangerous one (in terms of energy balance).

Table 9: Characteristics of the reactor 3	
Name of the system	HTC diving bottle experiment
Manufacturing company	-
Start of operation	-
Feed mode	Batch
Volume	12-15 Liter
Heating system	Open fire
Pressure range	Max pressure : 200-300 bar
Temperature range	?
Measurement system	None
Components	Pressure valve (sealed diving bottle)
Security system	None
Cost	No information
Contact	Christoph Koller
Address	Life Sciences und Facility Management Grüental, 8820 Wädenswil Phone: 058 934 56 25 E-Mail: christoph.koller@zhaw.ch
Remaks	Feedstock was fed through the tiny opening of the diving bottle and the bottle sealed. After reaction, it was difficult to empty the products from the bottle.

2.6.4 TFC engineering, Buchs

The plant will be located near a waste water treatment plant (WWTP) and an incineration plant in Buchs (SG). In the incineration plant, the dried fecal sludge is still wet and consumes a lot of energy to heat it until it can come to combustion. In an attempt to process the dried fecal sludge in a more efficient way, Roland Rebsamen (TFC engineering) designed this continuous HTC reactor to be used in combination with biowaste from a nearby composting plant. The process allows the heating value and the TS of the feedstock to be significantly increased compared to the usual dried fecal sludge (FS), which is advantageous with respect to the energy consumption for incineration (less heat required) and with respect to CO_2 emissions (less water content of the feedstock to be transported to the incineration plant).

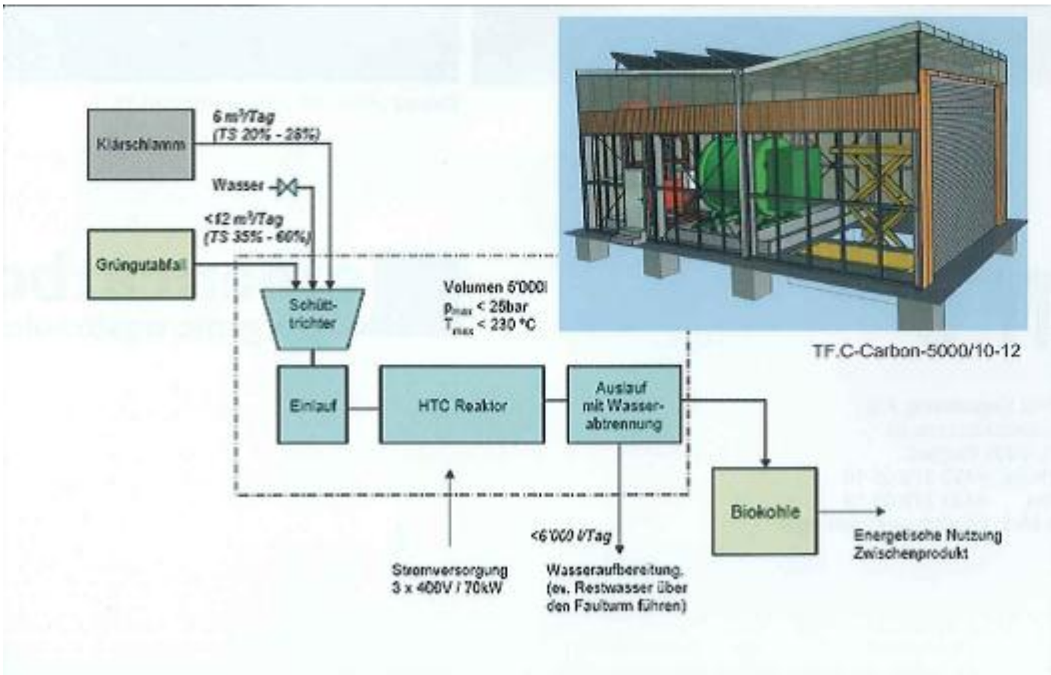


Figure 8: Schema of the HTC plant in Buchs (Source: TFC engineering leaflet)

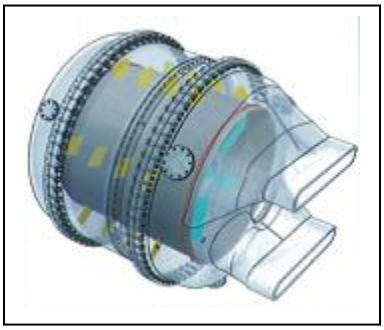


Figure 9: Schema of the reactor in Buchs (Source: TFC engineering leaflet)

Table 10: Characteristics of the reactor 4	
Name of the system	TF.C-Carbon-5000/10-12
Manufacturing company	TFC engineering, Kelag AG
Start of operation	Planned for December 2012
Feed mode	quasi-continuous; max capacity: 10 kton/year; retention time 3-4hours
Volume	5000 L
Heating system	Thermal oil mantle surrounding the reactor, oil heated with 50kW resistors, the oil is pressurized to provide adequate pressure to the compressible reactor.
Pressure range	20-25 bar
Temperature range	200-230°C
Measurement system	P,T sensors at input and output
Components	Reactor (stainless steel, composed of two tubes: inner for the inflow and outer for the outflow), Heating mantle (steel)
Security system	Exhaustion valves, reactor will be protected with grids, the users will have to wear helmet, face protection, gloves
Cost	3.5 M CHF
Contact	Roland Rebsamen

Address	TFC Engineering AG Industriestrasse 56 FL-9491 Ruggell Tel: 00423 / 375 05 10 Fax: 00423 / 375 05 19 E-Mail: info@tfc-engineering.li Homepage: www.tfc-engineering.li
Remarks	The reactor is designed to treat a combination of wet biomass (20 - 60% TS) composed of anaerobically digested sewage sludge from a WWTP (40%) and biowaste shredded in 2cm pieces (60%) as input materials. The cylindrical reactor lays on the side and rotates to avoid sedimentation; the feedstock is stirred mechanically inside. An elaborated heat recovery system allows the hot and pressurized output flow to preheat and pressurize the input flow. The input material is fed in a slit of a rotating cylinder made airtight with a graphite sealing system activated with oil. In the same way the output material goes in another rotating cylinder facing the input cylinder. When the two slits face each other, the heat exchange can take place. The start of operation of the plant was delayed due to technical problems with the operation of the reactor.

2.6.5 TFC engineering test reactor

Before starting to build the HTC plant in Buchs, Roland Rebsamen built a small HTC test-reactor, with which HTC tests were carried out. This is an example of a simple self-made reactor.

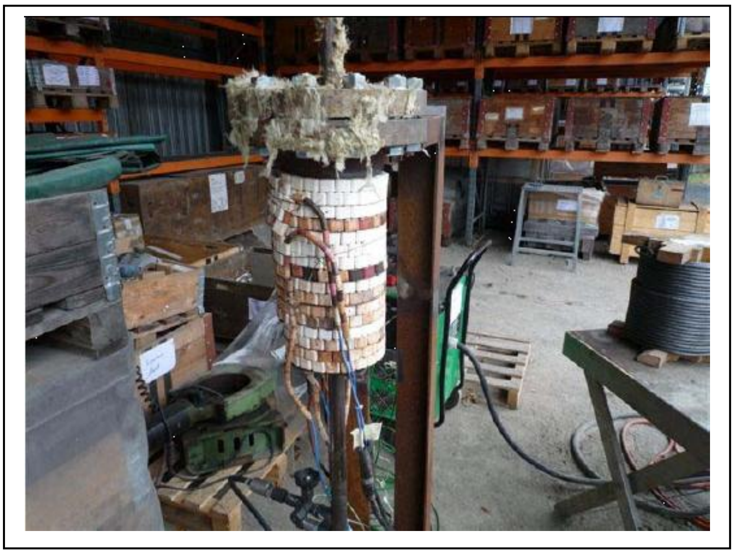


Figure 10: Picture of the test reactor (photo Rebsamen)

Table 11: Characteristics of the reactor 5	
Name of the system	TFC engineering test reactor
Manufacturing company	TFC engineering
Start of operation	-
Feed mode	Batch
Volume	About 20 L
Heating system	Electric mantle
Pressure range	< 25 bar
Temperature range	200°C
Measurement system	Pressure gauge
Components	Pressure vessel, heating mantle
Security system	Exhaustion valve
Cost	No information
Contact	Roland Rebsamen
Address	TFC Engineering AG Industriestrasse 56 FL-9491 Ruggell Tel: 00423 / 375 05 10 Fax: 00423 / 375 05 19 E-Mail: info@tfc-engineering.li Homepage: www.tfc-engineering.li
Short description	The reactor consists of a cylindrical pressure vessel closed with large screws. It doesn't possess any stirring system. No temperature sensor was used for the experiments; the temperature was set and determined with the heating mantle's regulating device.

2.6.6 AVA-CO2

Ava-CO_2 is a Swiss company that was founded in 2009. They design and build industrial scale HTC plant for factories aiming to revalorize their waste streams (organic waste, but also waste heat or steam). For this prospect they built a demonstration plant which is the worldwide first HTC plant working at industrial scale. The plant is located in Karlsruhe (Germany). In October 2012, the first commercial plant was planned to be brought to operation with 2 reactors working in parallel (multi-batch system) and 6 or 12 in a later phase. The main potential for the HTC-coal produced with these industrial plants is to use it as an energy carrier, for example in steel or cement factories or replacing dried sewage sludge in incineration plant.

Figure 11: Picture of the HTC plant in Karlsruhe (Source: AVA-CO2 leaflet)

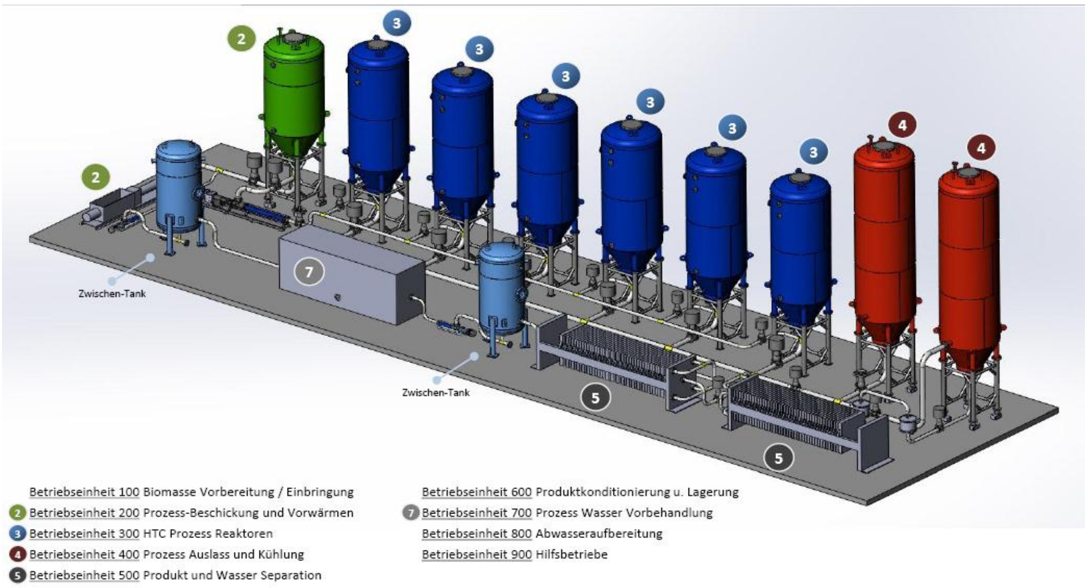


Figure 12: Schema of the AVA-CO2 system (Source: AVA-CO2 presentation at info day, October 2012)

Table 12: Characteristics of the reactor 6	
Name of the system	HTC-0
Manufacturing company	Ava-CO_2
Start of operation	October 2010
Feed mode	Batch, 3500 TS t/y, 2664 t/y HTC-coal produced (commercial plant), 5-10 hours retention time
Volume	No information
Heating system	Heat provided with steam (1.4 tons per batch)
Pressure range	22-26 bar
Temperature range	220-230°C
Measurement system	T,P sensors. Everything can be monitored from a separate room.
Components	Mixing tank, reactor(s), buffer tank, solid-liquid separation system, water treatment system

Security system	Interruption in case of overpressure, or clogging of the feedstock.
Cost	10-12 M Euros
Contact	Thomas Kläusli
Address	AVA-CO2 Schweiz AG Baarerstrasse 20 6304 Zug Switzerland Tel: +41 41 727 09 70 Mob: +41 78 936 74 81 Email: tk@ava-co2.com www.ava-co2.com
Short description	The plant can be operated with different types of feedstock (25-70% TS): sewage sludge, digestate from anaerobic digestion process, garden waste, organic fraction of municipal solid waste. The input material has to preprocessed such that it can be pumped in the reactor. The feedstock is first preheated in the mixing tank to 160°C, 10 bar. It is then pumped in the reactor where the HTC process takes place. It then goes in a buffer tank where it is cooled down and its heat recovered to preheat the next batch. The product is filtered and pressed for the production of HTC-coal. Part of the process water is recirculated, the rest goes through a filtration to be transferred to a WWTP.

2.6.7 Umwelt Campus Birkenfeld

In the framework of a research project, Moritz Mildenberger (Umwelt Campus Birkenfeld) designed and built a small HTC reactor. The reactor was designed to hold 60 bar and was validated by the TÜV. This is another example of a self-designed test reactor.

Figure 13: Picture of the reactor in Birkenfeld (photo Robbiani)

Table 13: Characteristics of the reactor 7	
Name of the system	Umwelt Campus Birkenfeld test reactor
Manufacturing company	Made by Moritz Mildenberger
Start of operation	No information
Feed mode	Batch
Volume	3 L
Heating system	Electrical mantle
Pressure range	<30 bar

Temperature range	180-250°C
Measurement system	T sensor, Pressure gauge
Components	Pressure vessel (stainless steel), heating mantle
Security system	Overpressure valve (30 bar), bursting disc (40 bar)
Cost	Material for pressure vessel : 1700 euro, Heating mantle: 200 euro Heat regulator: 300-800 euro Overpressure valve: 80 euro Bursting disc: 300 euro Temperature sensor : 80 euro Total: 2660-3160 euro
Contact	Moritz Mildenberger
Address	Labor für Hydrothermale Karbonisierung FH Trier, Umwelt-Campus Birkenfeld Postfach 1380 55761 Birkenfeld Tel: 06782-17-2648 Fax: 06782-17-1267 Email: m.mildenberger@umwelt-campus.de Url: htc.umwelt-campus.de
Short description	The reactor consists of a cylindrical pressure vessel closed with a flat end at the bottom and a flange on the top. It doesn't possess any stirring device. The product is generally emptied with a pump.

2.6.8 Cube of Destiny

Cube of Destiny is a project initiated by Erwin Wimmer (Initiative Zukunftsenergien). It consists of a box containing a HTC reactor to carbonize algal biomass. The idea is to use a 12V-battery to provide the energy required for the heating. The battery can then be charged through solar energy.

Figure 14: Picture of the Cube of Destiny (Initiative Zukunftsenergien website)

Table 14: Characteristics of the reactor 8	
Name of the system	Cube of Destiny
Manufacturing company	Initiative ZUKUNFTSENERGIEN e.V.
Start of operation	March 2013 (?)
Feed mode	Batch
Volume	5-10 L
Heating system	Electric mantle
Pressure range	10 bar
Temperature range	160-180°C
Measurement system	Thermostat
Components	Box containing: pressure vessel, heating mantle, battery
Security system	Temperature control
Cost	300-900 euros
Contact	Erwin Wimmer
Address	Initiative ZUKUNFTSENERGIEN e.V. Steinbühel 1 6410 Telfs / Österreich Telefon: +43/(0)660/2101425 E-Mail: info@zukunftsenergien.org
Short description	The reactor consists of a cylindrical pressure vessel sealed with 16 screws. It doesn't have any stirrer. A heat recovery system is planned to convert and store the excess heat in form of electricity using the Seebeck effect.

2.6.9 Agrokraft

Agrokraft is a company that is active in renewable energy projects in Germany. One of their fields of interest is the optimal utilization of waste streams, particularly from agriculture. Agrokraft developed a first pilot reactor to test the suitability of HTC for this purpose. They are planning the construction of a bigger pilot plant working in continuous mode. They see HTC as a promising technology to transform biomass in a decentralized way into a valuable product, and at the same time providing solution to the CO_2 problem.

Figure 15: Picture of the Agrokraft HTC system (Source: Agrokraft press release)

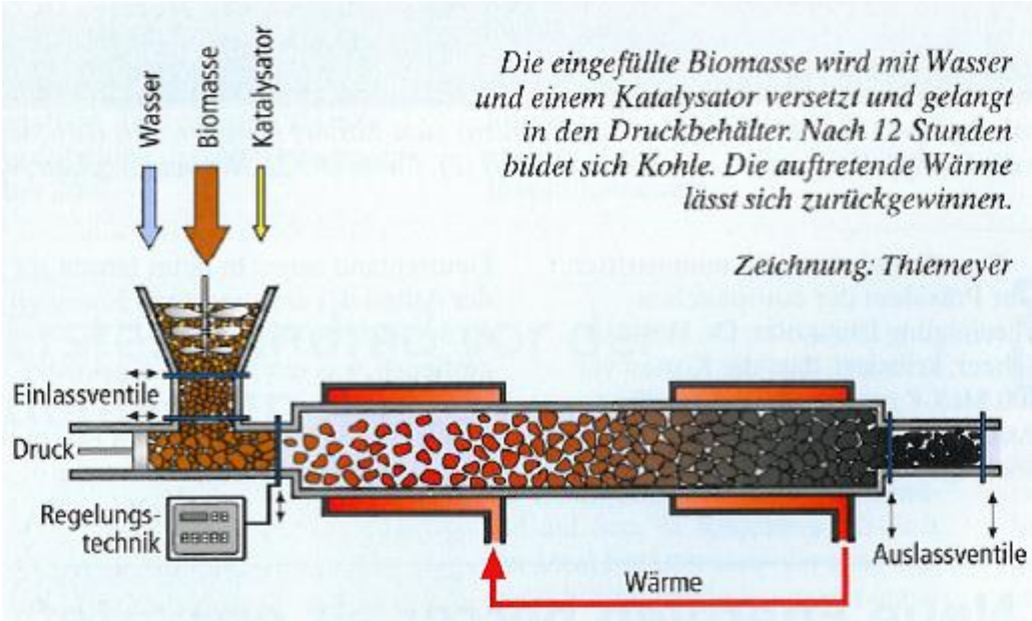


Figure 16: Schema of the Agrokraft HTC system (Source: Agrokraft press release)

Table 15: Characteristics of the reactor 9	
Name of the system	Mole
Manufacturing company	Agrokraft GmbH, Germany
Start of operation	2008
Feed mode	Continuous, 150 tons/year
Volume	150 Liter
Heating system	Heating oil mantle
Pressure range	circa 20-25 bar
Temperature range	180-200°C
Measurement system	No information
Components	7m long pressure vessel (structural steel), pumps. The reactor is lying horizontally and has a double mantle for the circulation of the heating oil.
Security system	Bursting discs in case of overpressure
Cost	50’000 euros
Contact	Michael Diestel
Address	Agrokraft GmbH Berliner Straße 19a 97616 Bad Neustadt/Saale Telefon +49 9771 6210-45 Telefax +49 9771 6210-49 info@agrokraft.de
Short description	A system of pressure gates takes the biomass to the pressurized reactor. The retention time vary between 4 and 16 hours before the watery mixture is released. As the plant was built with structural steel (and not stainless) its operation had to be stopped after 5 years.

2.6.10 Loughborough University

In the frame of the Reinvent the Toilet Challenge from the Bill and Melinda Gates Foundation, a team from the Loughborough University developed a toilet system using a HTC reactor to convert the fecal material to a safe coal-like material. This system is designed to be self-sufficient in terms of energy input. It is a continuous system based on a plug flow reactor design. Since it's still in the testing phase for the competition, some technical information could not be obtained.

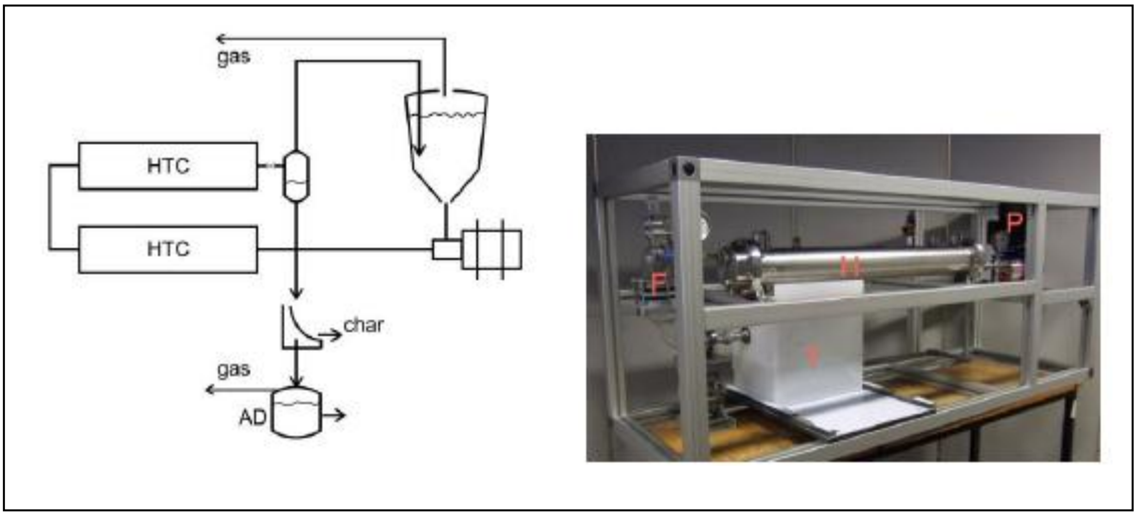


Figure 17: Schema and picture of the HTC reactor in Loughborough (Source: Danso-Boateng, et al., 2012)

Table 16: Characteristics of the reactor 10	
Name of the system	Loughborough RTTC
Manufacturing company	Loughborough University
Start of operation	No info
Feed mode	Continuous
Volume	No info
Heating system	No info
Pressure range	No info
Temperature range	No info
Measurement system	No info
Components	Collection tank, macerating pump, multi-pass reaction vessel, flash evaporation system
Security system	No info
Cost	No info
Contact	Professor M. Sohail (Khan)
Address	Civil and Building Engineering Loughborough University T: 01509 222890 E: M.Sohail@lboro.ac.uk

Short description	The waste material (urine, feces, and flush water) is pumped from the collection tank into the reactor vessel. The flow rate is set so that the material spends about 15 minutes inside the reactor vessel. After this the pressure is released in a flash vessel. A portion of the water immediately turns into steam taking with it volatile organic compounds. This steam is collected and used to pre-heat the material in the storage tank. After passing through the collection tank the remaining gas is collected and as it contains volatile organic compounds it can be fed back into the fuel stream. The liquid/solid stream from the flash vessel is filtered using a microslot filter. The solids are collected either for use as a fuel source or as a soil enhancing agent. The remaining liquid will contain a variety of low molecular weight carbon based compounds. This liquid will be treated in an anaerobic digester in order to generate methane fuel (Danso-Boateng, et al., 2012).

2.6.11 Summary

The different reactors were classified in a double axis diagram according to their size scale and their technological complexity. For the size three different categories were chosen:

Industrial scale: a plant that is big enough to work at a commercial level. (Volume > $1m^3$)

Bench scale: a plant that is built for research purposes to demonstrate the feasibility of a technology, but doesn't work at a commercial level (Volume > 3 Liters).

Lab scale: reactors which are smaller than 3 Liters and are used for research purposes are considered here as lab scale reactors.

Concerning the technological complexity, the reactors were attributed an ordinal rating between 1 and 5 (1 for low-tech and 5 for high-tech).

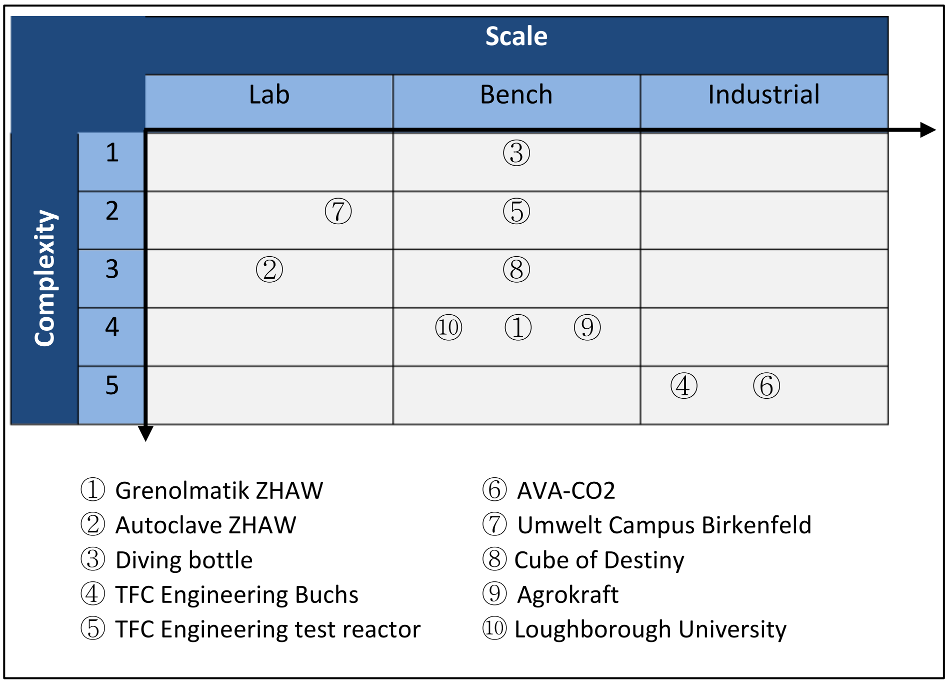


Figure 18: Classification according to scale and complexity of the different reactors

3. DESIGN SELECTION

3.1 DESIGN REQUIREMENTS

3.1.1 Size and complexity of the reactor

The reactor should be adapted to conditions in developing countries and therefore should have a simple design and made of low-tech components. It is planned to be used for experiment purposes and designed such that it can be operated by one single person (weight and size constraint). The reactor should be big enough to produce a significant amount of HTC-coal such that the use of the products can be maximized. A large reactor also has the advantage that the relative error on the measurements of the quantity of input and output material is minimized (larger recovery ratio and diminution of losses proportionally). However in case of a cylindrical reactor, a large diameter implies more heat dissipation through the upper surface and the bottom of the reactor, which means a longer period of time to bring the inner temperature to the desired level, especially if the reactor is not stirred.

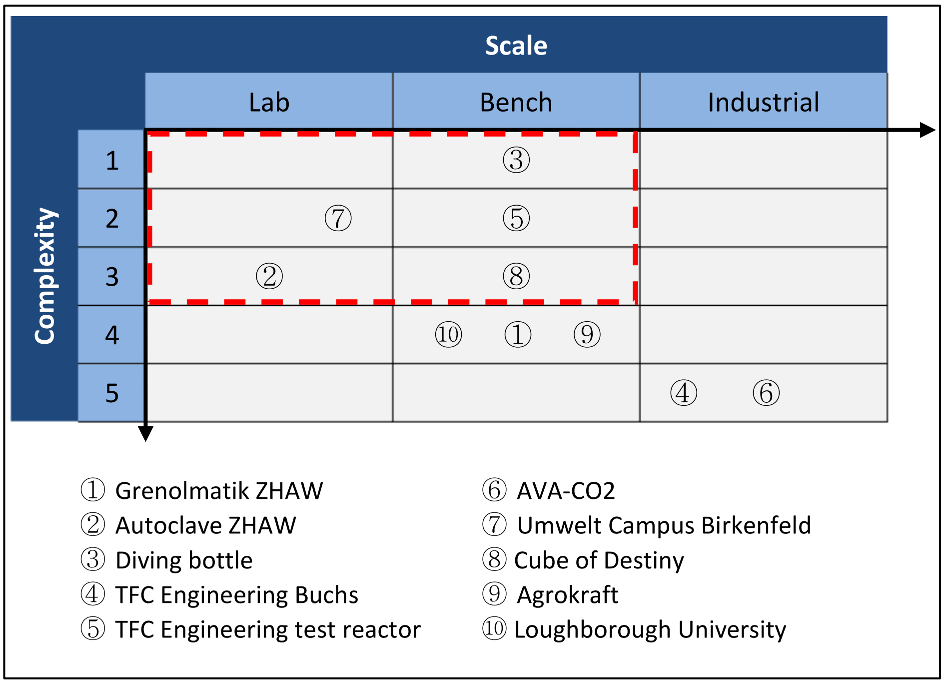


Figure 19: Classifications of the reactors with red rectangle indicating the scale and complexity ranges of interest

3.1.2 Regulation and standard for the construction and design of pressure equipment

Since May 2002, in Switzerland and the EU, the Pressure Equipment Directive (PED) 97/23/EC is applied for the design, construction and conformity assessment of all pressure equipment having a volume of more than one liter and a maximum pressure of over 0.5 bar [6]. The reactor is thus subject to the directive and has to be designed, constructed and tested in compliance with it.

This regulation requests all pressure equipment and assemblies placed in circulation[2] and put in service: “to be safe, to meet essential safety requirements covering design, manufacture and testing, to satisfy appropriate conformity assessment procedure and carry the CE-marking (European Conformity mark)” [7,8].

To ensure the design and construction of pressure equipment to be conform to this regulation, some code of practice have to be followed. For example AD-2000 is a standard made by the German Pressure Vessel Association (Arbeitsgemeinschaft Druckbehälter) and is frequently used in Germany and Switzerland. Another example is the European standard EN 13445. Internationally (outside the European Union) the American ASME-code from the American Society of Mechanical Engineers is the one that is usually accepted [9].

3.2 Possible options of feeding for the reactor

HTC plants can be classified in two different feeding-mode categories: continuous or batch, depending if the feedstock is fed into the reactor continuously or batch-wise.

3.2.1 Batch

Batch reactors are usually cylindrical stirred tanks. They can be filled with any type of organic feedstock. It is only when the reactor is filled that the carbonization process starts. Once it is over, the reactor is emptied before being loaded with new material. To optimize the process, industrial plants using this system usually operate various reactors in parallel (quasi-continuous multi-batch system). In this way, the feedstock can always be fed in one of the reactors without waiting for the reaction to be over. Moreover the waste heat from a reaction can be reused to preheat the input material for the next reaction.

3.2.2 Continuous

Continuous reactors are usually smaller than batch reactors. They require a more elaborated system to handle the feedstock as a flowing stream while maintaining a high pressure in the reactor. This can be done for example with a screw pump displacing the feedstock along the

[2] Als Inverkehrbringen gilt die entgeltliche oder unentgeltliche Übertragung oder Überlassung von Druckgeräten und Baugruppen. Etwas gilt als übertragen oder überlassen, sobald es der Benutzerin oder dem Benutzer erstmals zur Verfügung steht.

screw's axis. A system of locks where the biomass is brought step by step to a higher pressure can also be used. An alternative is to mix the biomass with water, with high water content such that the mixture can be pumped and brought in the reactor with a spray nozzle (Krause, 2010). This system allows the reactor to stay continuously at the same temperature, without the need to be cooled down and reheated in between two reaction cycles. Furthermore, the heat of the output material can be partly recovered by directly preheating the input material with a heat exchanger.

3.2.3 Comparison of the different options

Table 17: Comparison of batch and continuous mode for HTC systems (Modified from Krause, 2010)

Characteristics	Batch	Continuous
Level of development	Demonstration plant	Mostly pilot plants, also industrial plant
Complexity	Low	Middle to high
Handling of feedstock	Manual or mechanical	Mechanical
Heat recovery	Indirect heat recovery from process water	Direct heat recovery from output stream
Feedstock preprocessing	To allow easy stirring and pumping (in case of mechanical operation)	Required for the handling of the flowing stream against reactor pressure
Advantages	Simple process, easy process control	High energy efficiency
Disadvantages	Require bigger reactor for the same production rate	Feeding of feedstock against reactor pressure, need of electricity for mechanical handling

3.3 Possible options for the heating system

For the heating system, three different options are considered.

3.3.1 Thermal oil mantle

The reactor is surrounded by a double mantle or a piping system. Thermal oil is heated at the required temperature and flows through the closed loop system. Temperature of the oil can commonly reach up to 350°C while the pressure remains low. The energy needed to heat the oil can be provided for example by combustion of a fuel (oil, gas), by electrical heating or with concentrated solar radiation. This system requires an automated temperature regulation system that controls the oil temperature and avoids it to be too high or too low.

3.3.2 Electric mantle

An electric mantle made of electrical resistors surrounds the reactor. Heat is produced through Joule effect when an electric current passes through the resistors. The temperature is fixed by a simple thermostat. In case the temperature goes too high, the current supply is switched off. An isolating material needs to be used to surround the reactor to avoid the dissipation of energy in the surrounding environment. For a current supply independent of the grid, it can be combined with photovoltaic panels.

3.3.3 Steam

Steam is produced in a boiler and then heated further in a superheater at saturated steam conditions (high pressure and high temperature). Heat is provided to the HTC reactor by injecting the high temperature steam in the reactor, which also allows the content to be stirred. Heat for the boiler can be provided by the combustion of any type of fuel (wood, coal, oil natural gas), by electric heating, by concentrated solar radiation or by using waste steam from other processes.

3.3.4 Comparison of the different options

Table 18: Comparison of the different heating systems

Characteristics	Thermal oil mantle	Electric mantle	Steam
Main advantages	Safe and simple operation	Cheap and simple system	Steam injection allows stirring of the feedstock
Main disadvantages	Complex and expensive heat regulation system	Energy losses	Pressurized steam may represent security risks, more fitting and apparatus required

3.4 SELECTION OF AN APPROPRIATE DESIGN

The design selection of the HTC prototype reactor is made according to certain criteria. These criteria have been identified to help selecting an option that is adapted to conditions in developing countries.

3.4.1 Criteria for application in developing countries

Cost: the selected option should be made of low cost material. The design shouldn't involve the use of expensive equipment.

Availability: the different materials should be available in developing countries.

Level of technology: the selected option should be easily reproducible, the design simple, and it should be easily constructed (no experts needed).

Durability: the equipment should be able to be used in the long term, without the need of frequent maintenance or troubleshooting.

Ease of handling: the operation and maintenance of the equipment shouldn't need complex infrastructure, and/or expert knowledge.

Security: the selected option should allow for a safe operation of the reactor.

3.4.2 Evaluation table

On the left of this table, the selection criteria are listed. For each option, a value between 1 and 5 is attributed to help determining for which one of the 3 options the criteria are the most fulfilled (1 = not fulfilled, 5 = fulfilled). The options with the most points will be considered as being the most appropriate.

Table 19: Evaluation table of the different options

Criteria	Feed-mode		Heating system		
	Continuous	Batch	Electric	Oil	Steam
Cost	2	4	5	3	2
Availability	2	4	3	2	2
Level of technology	2	4	4	4	2
Durability	3	4	4	4	2
Ease of handling	3	4	4	3	1
Security	3	3	4	4	1
Total	**15**	**23**	**24**	**20**	**10**

The evaluation table shows that the option that fulfills the criteria best is the batch reactor heated by means of an electric mantle.

4. DESIGN

4.1 REACTOR SPECIFICATIONS

4.1.1 Description of the reactor

The batch reactor will consist of a pipe closed to one end with a vessel dished end (curved shape). This requires less material than a flat end and is easier to manufacture than a hemispherical end. The top is equipped with a flange and closed with a lid that can be screwed to the flange, allowing easy accessibility to the inside of the reactor. This way, the reactor can be easily opened, filled, and tightly closed. A graphite sealing ring allows the reactor to be hermetically sealed.

The electric heating is provided by a cylindrical heating mantle surrounding the vessel. The external temperature is controlled with a regulator connected to the heating mantle. An energy meter is connected to the heating mantle to measure the energy consumed during the reaction. The inner temperature and pressure as well as power consumption will be recorded over time on a computer during the reactions. The maximum allowable pressure is controlled with an overpressure valve that releases the pressure when going higher than a certain limit. The steam released is directed to the outside with a stainless steel pipe. At the end of the reaction, after letting the reactor cooling down, the residual pressure will be released thanks to a drain valve and the residual gases directed to the outside through a plastic pipe.

After the reaction is completed, the content of the reactor needs to be recuperated. The reactor can be fixed with two lateral rods on a frame with bearings from which it can rotate. This simple rotating system allows the content to be easily emptied and the reactor washed after every reaction. Another possibility would be to use a separate internal container that can be easily removed from the reactor and easily emptied after the reaction. The disadvantage of such a system would be that it increases the thickness between the heating mantle and the substrate, worsening the heat transfer between the two.

A removable transversal bar fixed to the inferior rod at the bottom of the reactor allow the rotation to be blocked when needed (for example while opening and closing the reactor or during the reaction). An additional hole is also provided on the lid, leaving the possibility to change the disposition of the measuring instruments (for example inner temperature measured at the side rather than in the middle) or to add a new device (pH-meter, sampling valve, stirrer).

The possibility to install a stirrer has also been investigated. Three possible types of stirrer were identified. Magnetic agitator (20'000.- euro), magnetic coupled stirrer (40'000.-), stirrer with a (rotating) mechanical shaft-seal (no information could be obtained about the exact price but at least 10'000.- is estimated for such a device).

Since such a system involves elevated costs and significantly increased overall technological complexity of the reactor, the possibility of not using a stirrer was considered. One consequence might be that a longer retention time is needed for the feedstock to reach the required carbonization conditions in comparison with a system where the substrate is stirred. Another possible consequence is that the end product could be rather inhomogeneous.

Experiments were conducted in collaboration with ZHAW (using the Grenolmatik HTC reactor) in order to inquire about possible differences between HTC of biomass with and without stirrer. The results showed no significant differences of the HTC-coal and process water characteristics (See results of the experiments in the Appendix). Thus it was decided not to implement a stirrer to the prototype reactor.

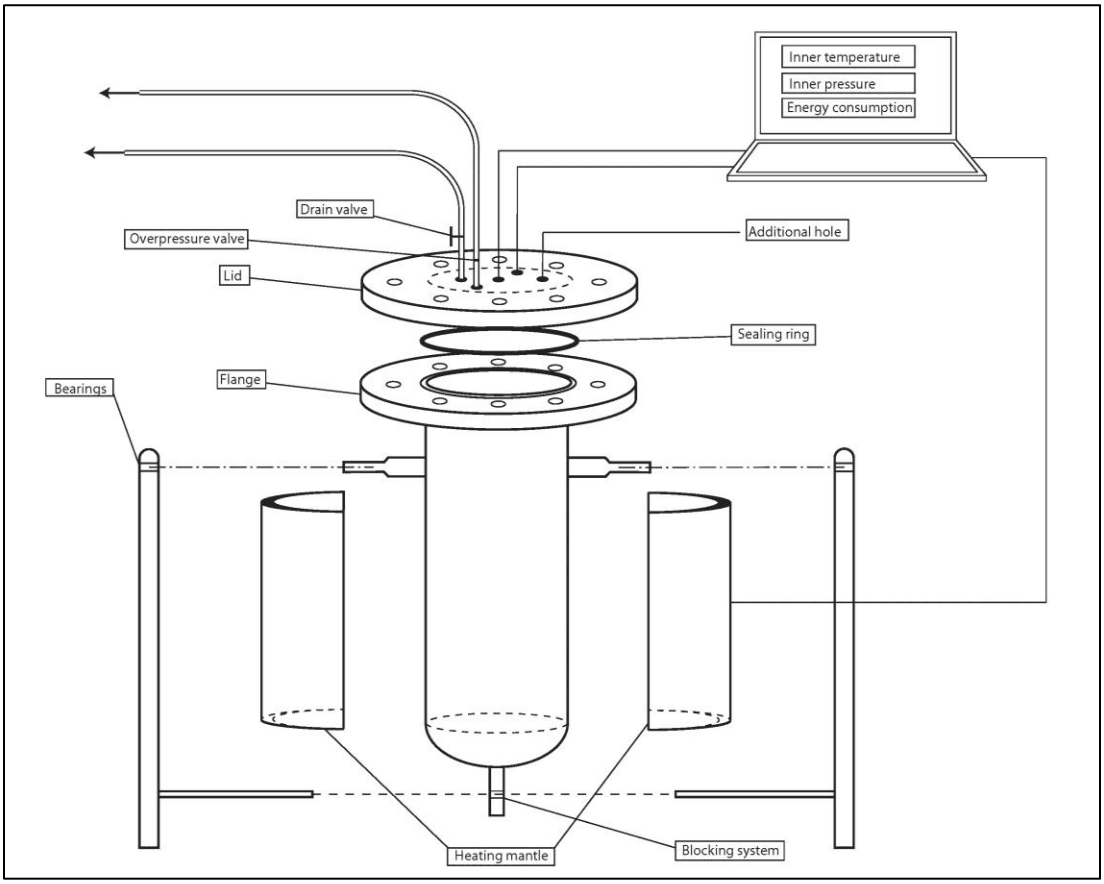


Figure 20: Schema of the prototype reactor

4.1.2 Size

Pipes for the wall of the reactor can be found in different standard sizes which conform to International Standard Organization usage. The size of pipe is designated by the acronym DN (diameter nominal) [5].The size of pipe chosen is DN 200 which means an inner diameter of 200 mm. This choice was inspired by the Grenolmatik HTC-reactor at ZHAW. The batch reactor is planned to have a capacity of about 20 liters (this means a pipe 600 mm long).

4.1.3 Carbonization conditions and maximal conditions

Organic compounds such as sewage sludge or biowaste are carbonized at temperatures between 180 - 220°C with resulting pressures of 10 - 25 bar [4]. For determining the maximal design temperature, not only the temperature of the feedstock has to be taken into account but also the highest possible temperature of the material. When heating with an electric mantle, the walls of the reactor in contact with the heating mantle necessarily have a higher temperature than its content. The lower the heat transfer between the mantle and the substrate, the higher the temperature attained at the walls [9]. The maximum allowable temperature is then set to the maximal value that can be attained with the heating mantle, which means 300°C. Regarding the pressure, the maximum allowable pressure is set to 30 bar, which allows for a 5 bar margin.

4.1.4 Lifetime

For the design of the reactor, a lifetime n has to be specified. It represents the number of pressure cycles within which the reactor is certified to be operated safely. A pressure cycle is defined as the number of time the operating pressure is reached starting from the conditions rest. Pressure equipment subject to more than 1000 load cycles requires specific additional calculations [8].

4.1.5 Classification

The classification is made according to the pressure, volume and fluid group. Since the gas phase in the reactor may contain methane, the fluid group category is designated as inflammable and thus dangerous (group 1). With this type of substance, the reactor is classified under category III of the PED 97/23 EC. This requires appropriate materials and welding work, qualified welders, non-destructive testing, construction drawings and calculations, a risk analysis and a user manual. Materials, design and construction are subject to a conformity assessment and must be certified by an entitled authority [12]. The reactor will be designed according to AD2000-standards.

4.1.6 Summary

Table 20: Summary of reactor specifications	
Applied regulation	PED 97/23 EC - AD2000
Fluid group	1 (inflammable)
Category	III
Pipe size (Diameter)	DN200
Volume	20 Liters
Pressure range	10-25 bar
max allowable pressure	30 bar
Temperature range	180-220°C
max allowable T	300°C
Maximum number of load cycles	1000

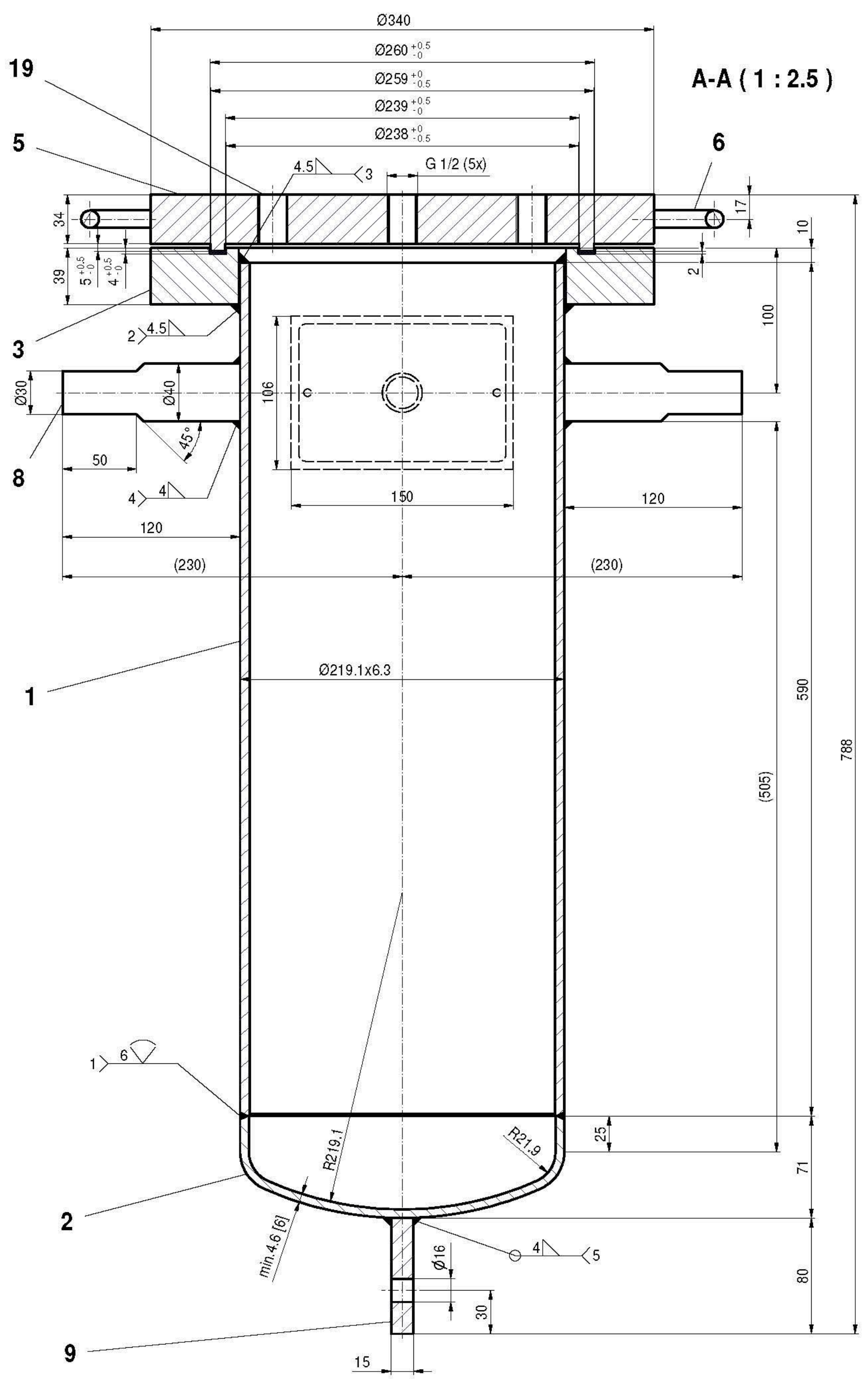
4.2 MATERIALS

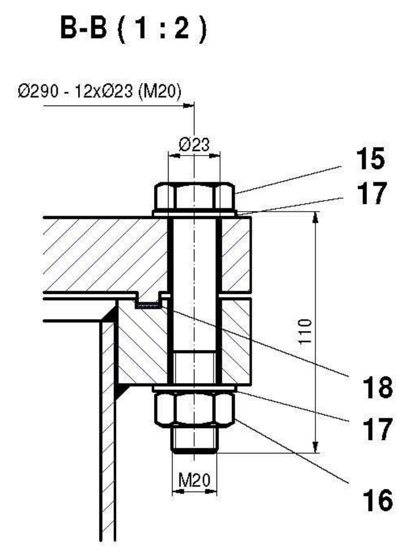
The pressure tank, which is in contact with water and should be resistant to acidic conditions, has to be made of stainless steel. Stainless steel is a low carbon steel that contains chromium (Cr) with a minimum of 10% of mass content which gives it its stainless, corrosion resisting properties. Stainless steels can be divided into three categories according to their crystalline structure: austenitic, ferritic and martensitic. Austenitic steels have excellent corrosion and heat resistance with good mechanical properties over a wide range of temperatures. The most widely used contain Chromium and Nickel (Ni). Other elements such as molybdenum (Mo) and titan (Ti) can also be present depending on the grade [1,3].

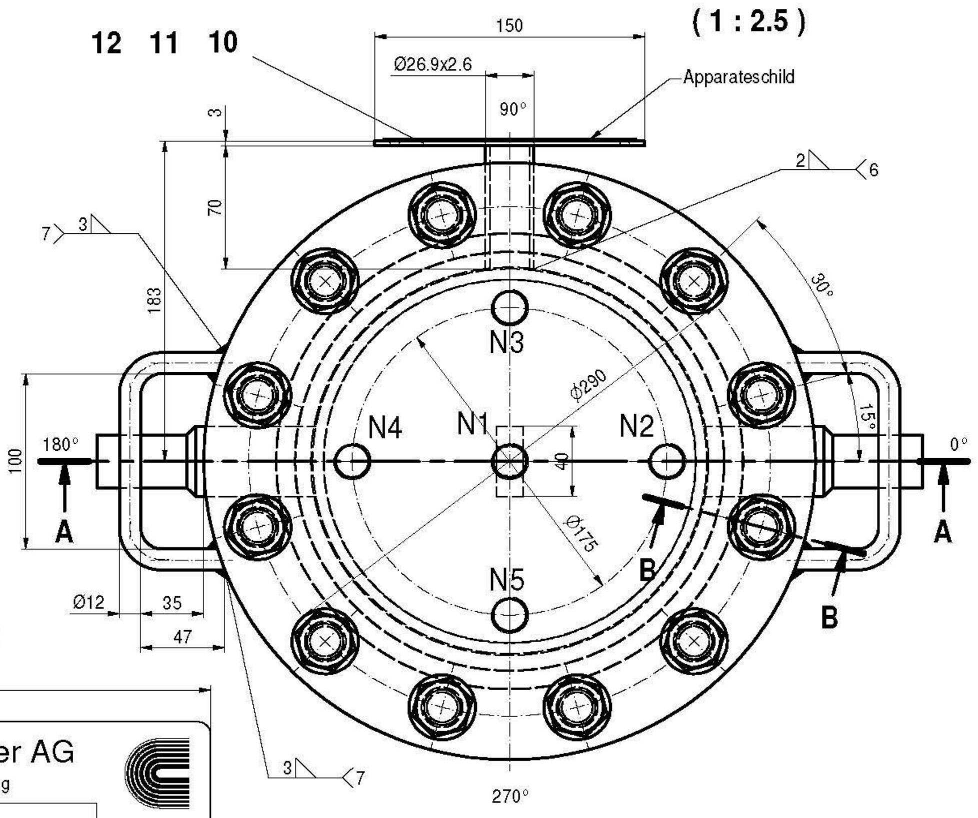
Table 21: Description of the different austenitic stainless steels

Steel Name	Steel Number	SAE Steel Grade	Description (source: [2])
X5CrNi18-10	1.4301	304	Most versatile and widely used stainless steel
X2CrNi18-9	1.4307	304L	Low carbon version of 304 to increase weldability
X6CrNiTi18-10	1.4541	321	Similar to 304 but lower risk of weld decay due to addition of titanium
X5CrNiMo17-12-2	1.4401	316	Contains an addition of molybdenum that gives it improved corrosion resistance
X2CrNiMo17-12-2	1.4404	316L	Low carbon version of 316
X6CrNiMoTi17-12-2	1.4571	316Ti	Contains a small amount of titanium for heat resistance

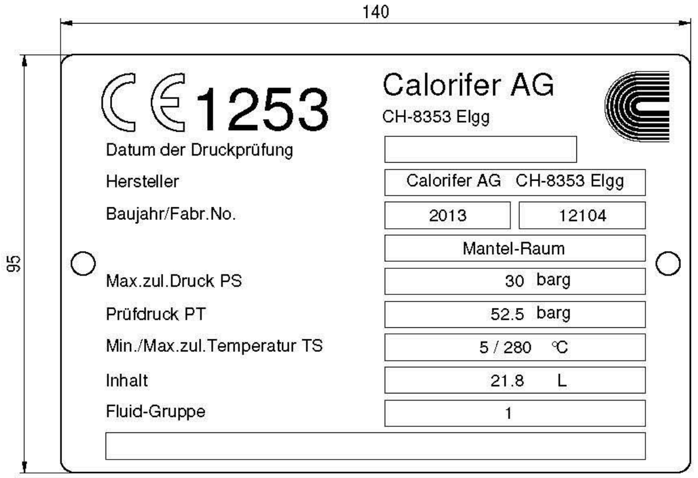
4.3 DESIGN AND DIMENSIONS OF THE REACTOR





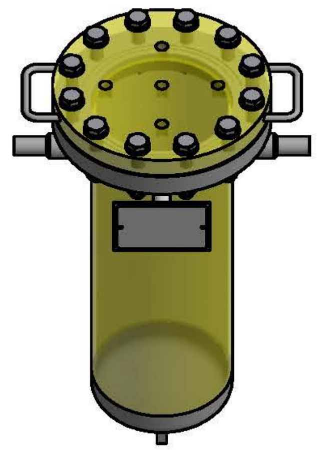


Apparateschild M1:1



Oberflächenausführung:
Innen und aussen gebeizt und passiviert.

Schweissnahtnummerierung:
Behälter: 1-3
Anschweissteile: 4-7



Stutzentabelle / Nozzles Table

Nr.	PN	DN	Rohr / Tube ø	Norm / Standard	Funktion / Function	Bemerkung / Remark
N1	-	G 1/2	-	ISO 228	Gewindeanschluss	mit Dichtring aus SS
N2	-	G 1/2	-	ISO 228	Gewindeanschluss	mit Dichtring aus SS
N3	-	G 1/2	-	ISO 228	Gewindeanschluss	mit Dichtring aus SS
N4	-	G 1/2	-	ISO 228	Gewindeanschluss	mit Dichtring aus SS
N5	-	G 1/2	-	ISO 228	Gewindeanschluss	mit Dichtring aus SS

Q0440612-201	SP0440612	PO0440612	B0440612-201	PN-16606
QM-Datenblatt QM-DATASHEET NO.	Schweissvorschrift WELDING SPECIFICATION NO.	Bauprüfplan INSPECTION & TEST PLAN NO.	Stückliste PARTS LIST NO.	Projekt-Nr. PROJECT NO.

Rev. REV.	Änderungen MODIFICATION	Datum / DATE Gez. / DRAWN	Visum VISA	Datum / DATE Gepr. / APPROVED	Visum VISA
2	Haltegriffe	21.02.2013	harra	21.02.2013	harra
0		01.02.2013	harra	05.02.2013	harra

Allgemeintoleranzen: / GENERAL TOLERANCE: EN ISO 13920, DIN 28005, DIN 28008-LT

Normal / Standard Projektion DIN

Titel / TITLE: **HTC Reaktor**
DN200

Separate Stückliste / SEPARATE PARTS LIST: ja/yes

Massstab / SCALE: 1:2.5

Stutzenabstände bei Normflanschen beziehen sich auf Dichtfläche.

NOZZLE DISTANCES OF STANDARD FLANGES REFER TO GASKET SURFACES.

CALORIFER
CH-8353 Elgg

K / W | Blatt / SHEET: 1 | von / OF: 1 | Format / FORMAT: A2 | Zeichnungsnummer / DRAWING NUMBER: B0440612 | Rev. / REV.: 2

Table 22: List of the different parts of the reactor					
Item	**Quantity**	**Designation**	**Dimension [mm]**	**Material**	**Weight [kg]**
1	1	Cylindrical shell	219.1 x 6.3 x 590	1.4571	19.5
2	1	Vessel dished end	219.1 x min.5.4 x ED 6	1.4571	3
3	1	Flange	39 x ø221 x ø340	1.4541	14.3
5	1	Closure head	39 x ø340	1.4541	22.8
6	2	Handle	12 x 202	1.4404	0.4
8	2	Round bars	40 x 120	1.4404	1.9
9	1	Flat bar	40 x 15 x 80	1.4404	0.4
10	1	Tube	26.9 x 2.6 x 70	1.4571	0.1
11	1	Support plate	150 x 106 x 3	1.4307	0.4
12	1	Identification plate		1.4301	0.1
15	12	Hexagonal screw	M20 x 110	A2-70	4.2
16	12	Hexagonal nut	M20	A2-70	0.8
17	24	Washer	M20 x 37/21 x 3	A2	0.4
18	1	Flat cylinder head seal	259 x 239 x 2	Graphite	
19	5	Sealing ring	26 x 21 x 1.5	1.4571	
Total weight [kg]					68
Total cost [CHF] (to which must be added around CHF 6000.- for the certification)					5'700.-

4.4 ADDITIONAL EQUIPMENT AND MEASURING INSTRUMENTS

Table 23: List of additional equipment and measuring instruments				
Designation		**Characteristics**	**Description**	**Cost [CHF]**
Additional equipment	Overpressure valve SV510 Spyrax Sarco AG	Maximal pressure: 30 bar Connection: G1/2'' Maximal temperature : 280°C	Security valve which releases the pressure when the maximal allowable pressure is reached.	600.-
	Drain valve AV243 Spyrax Sarco AG	Connection G1/2'' Max temperature: 400°C Max pressure: 300 bar	Valve to release the residual pressure once the reaction is over.	220.-
	Heating mantle HFH Temperature regulator HT42-30P Hillesheim GmbH	Diameter: 219 mm Length: 500 mm Maximal temperature: 300°C Maximal Power: 2500 W	Isolated electric mantle with internal temperature sensor and regulator	1570.-
Measuring instruments	Temperature sensor W120.3L Display ACS 13A Roth + CO. AG	Sensor type: PT 100 Connection: G1/2'' Sensor length: 250 mm	Sensor connected to a display from which the data can be transferred to a computer via USB cable.	920.-
	Digital manometer Leo Record Keller AG	Pressure range: 0-31 bar Temperature range: 20-300°C Connection: G1/2''	Pressure sensor from which the data can be transferred to a computer via USB cable.	1680.-
	Energy meter VSM-120 VSM-102 counter VSM-101 Gateway Voltcraft	Current range: 0-80 A	Energy consumption logger, data transmission through USB radio stick	340.-

Since the maximum allowable temperature of the overpressure valve is 280°C, the maximum allowable temperature of the reactor has also been set to 280°C (instead of 300°C).

4.5 Costs

Table 24: Details of the costs

Item	**Cost [CHF]**
Reactor	5’700
Certification	6’000
Additional equipment	2’400
Measuring instruments	2’900
Total	**17’000**

For comparison, the cost of a state-of-the-art reactor of similar size, the Grenolmatik 25 (Grenol GmbH, Germany, see table 7) costs around 100’000 CHF.

5. CONSTRUCTION

The construction of the reactor was carried out by the apparatus manufacturing company Calorifer AG in Elgg (ZH). The inspection and the conformity assessment according to European Directives were carried out by a notified body third party called Swiss TS (Wallisellen ZH).

5.1 CONSTRUCTION AND CERTIFICATION PROCEDURE

Prior to manufacturing the company Calorifer AG did a hazard analysis for the HTC reactor and checked its concept. They checked the design and calculated the precise dimensions in compliance to the AD-2000 directives. The design and dimensions were then sent to Swiss TS for approval. Once approved, the different parts of the reactor could be purchased or fabricated. The material certificates of the different parts were checked by Swiss TS as well as the certificates for procedure qualification record (PQR), welding procedure specification (WPS) and welder performance qualification (WPQ). Once approved, the welding works could be carried out.

Seven different weld seams had to be welded (numbered from 1 to 7 as in the technical drawing above). Weld seams 1,2 and 3 bind together the elements of the reactor which will be exposed to the high pressure. They are therefore thicker than the others and have to undergo a nondestructive examination. The others (4,5,6 and 7) bind other elements such as the identification plate, the handles, or the lateral and inferior rods to the reactor and are not exposed to the high pressures. Weld seam 1 is of V shape (see figure) and binds together the vessel dished end with the cylindrical shell. It is welded with GTAW. Weld seams 2 and 3 bind the flange to the cylindrical shell and are of type F. Weld seam 2 was welded with SMAW whereas weld seam 3 was welded with GTAW.

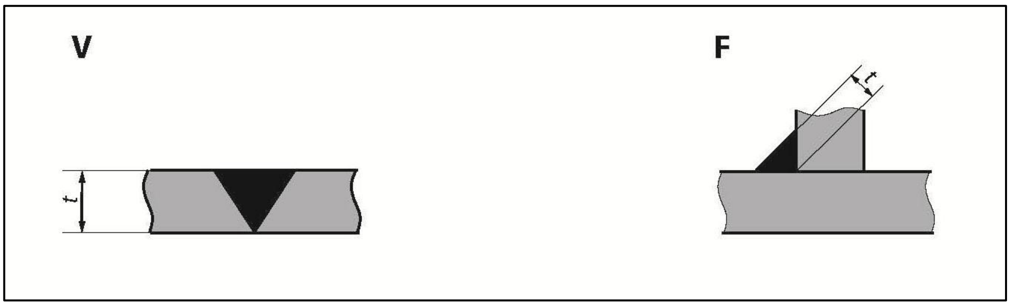


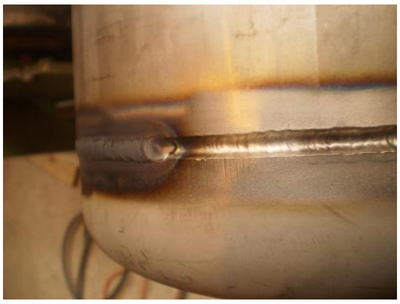
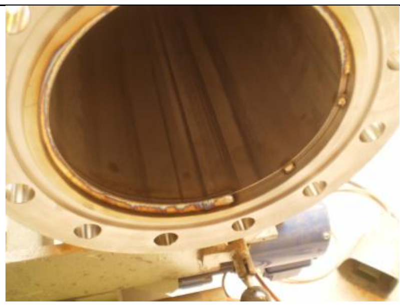
Figure 21: Shapes of weld seams

GTAW (Gas Tungsten Arc Welding) and SMAW (Shielded Metal Arc Welding) are two welding techniques in which an electric arc is created between an electrode and the metal. The high temperature generated allows the metal to melt at the welding point. With GTAW the electrode is made of Tungsten and is not consumed during the welding. An inert gas is used to avoid the contact between the metal in fusion and the atmosphere. With SMAW, a consumable electrode is used to lay the weld. The electrode is coated with a material that melts during the welding which provides shielding gases and a layer of slag to protect the weld area from atmospheric contamination.

After the construction, the weld seam 1 was examined with Radiographic Testing and the weld seams 2 and 3 were examined with Liquid Penetrant Testing. These are two different methods for the examination of weld seams: Radiographic Testing (RT) uses penetrating electromagnetic radiation such as X-Rays to detect defects in the weld seam and Penetrant Testing (PT) method uses a visible dye that is directly applied on the weld seam. Swiss TS then inspected visually and approved all the weld seams as well as the inside of the reactor.

Table 25: Specification of weld seams

Weld N°	Material 1	Material 2	Shape	Thickness *t* [mm]	Nondestructive examination	Weld procedure
1	1.4571	1.4571	V	6.0	RT	GTAW
2	1.4571	1.4571	F	4.5	PT	SMAW
3	1.4571	1.4571	F	4.5	PT	GTAW
4	1.4571	1.4404	F	4.0	-	GTAW
5	1.4571	1.4404	F	4.0	-	GTAW
6	1.4571	1.4571	F	2.0	-	GTAW
7	1.4541	1.4404	F	3.0	-	GTAW

Table 26: Pictures of weld seams		
Weld N°	**Pictures during welding work**	**Pictures of welds seams during welding**
1		
2		
3		

Finally, a hydraulic pressure test was carried out under the supervision of Swiss TS. Water at 52.5bar/25°C was introduced in the reactor and maintained during at least 30 minutes without drop of pressure. This high pressure had to be applied to imitate the stress endured by the material at the maximal allowable pressure and temperature of the reactor (30bar/280°C).

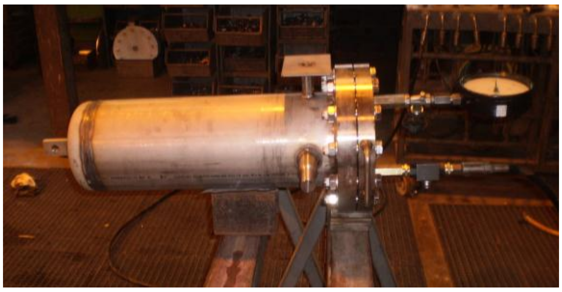


Figure 22: Picture of the reactor during the hydraulic pressure test

Once the test passed, the reactor was marked with a CE-label with the above mentioned characteristics. This certifies that the reactor conforms to the requirements of the EC directives.

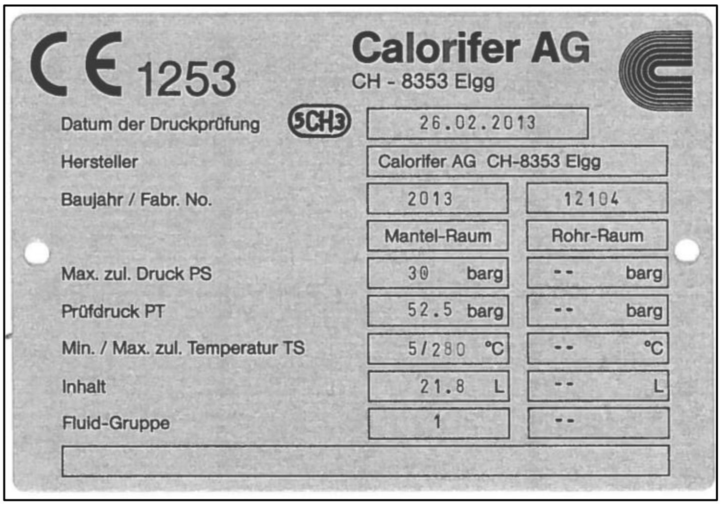
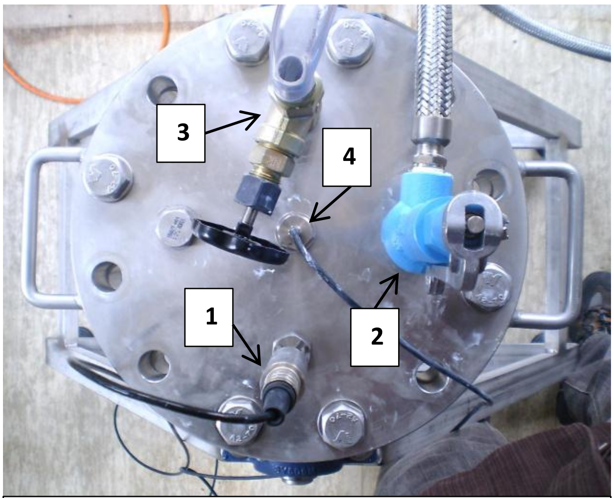


Figure 23: Identification plate with CE label

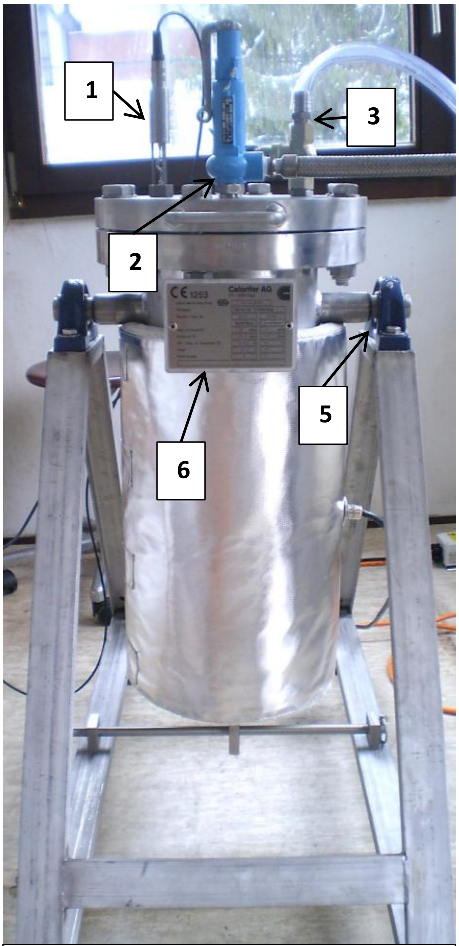
5.2 Final installation of the HTC reactor

The following pictures show the final system with the different measurement sensors (1 and 4) valves (2 and 3) as well as heating mantle. The overpressure valve is connected to a stainless steel pipe for the exhaustion of the possible steam released. The drain valve is opened only when the reactor is cooled down to release the residual pressure in the reactor. Thus the draining pipe is not exposed to high temperature steam and can be directed to the outside with a simple plastic pipe. The pictures show the frame with rotating system and bearings (5) as well as the blocking system. More picture of the instruments displays and connections as well as of the energy meter can be found in the next chapter.

N°	Description
1	Pressure sensor
2	Overpressure valve
3	Drain valve
4	Temperature sensor
5	Bearing
6	Identification plate with CE sign



View on the lid from top



Front view with heating mantle

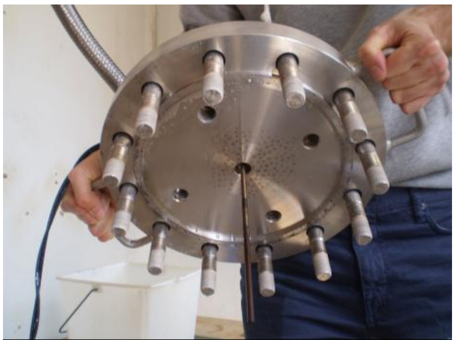
Figure 24: Pictures of the final installation

General view with stainless steel pipe for exhaustion of steam from overpressure valve

Lateral view without lid

Blocking system

Open reactor (with graphite sealing ring) after a reaction



Inferior part of the lid with temperature sensor and screws

Figure 25: Pictures of the final installation 2

6. TESTING OF THE REACTOR

To test the reactor, three experiments were carried out. The first experiment was carried out without substrate (only water). In the second and third tests, the HTC of rice was tested at different solid loads. Rice is a model substrate that can be easily carbonized under standard conditions. It is a convenient feedstock to use for testing as it can be easily obtained and is homogenous with regard to TS and elemental composition.

6.1 METHODS

6.1.1 Experimental set-up

Inner pressure and temperature as well as the energy consumption of the heating mantle are recorded during each process. The external temperature is imposed with the regulation device of the heat mantle. Inner temperature is measured at the center of the reactor.

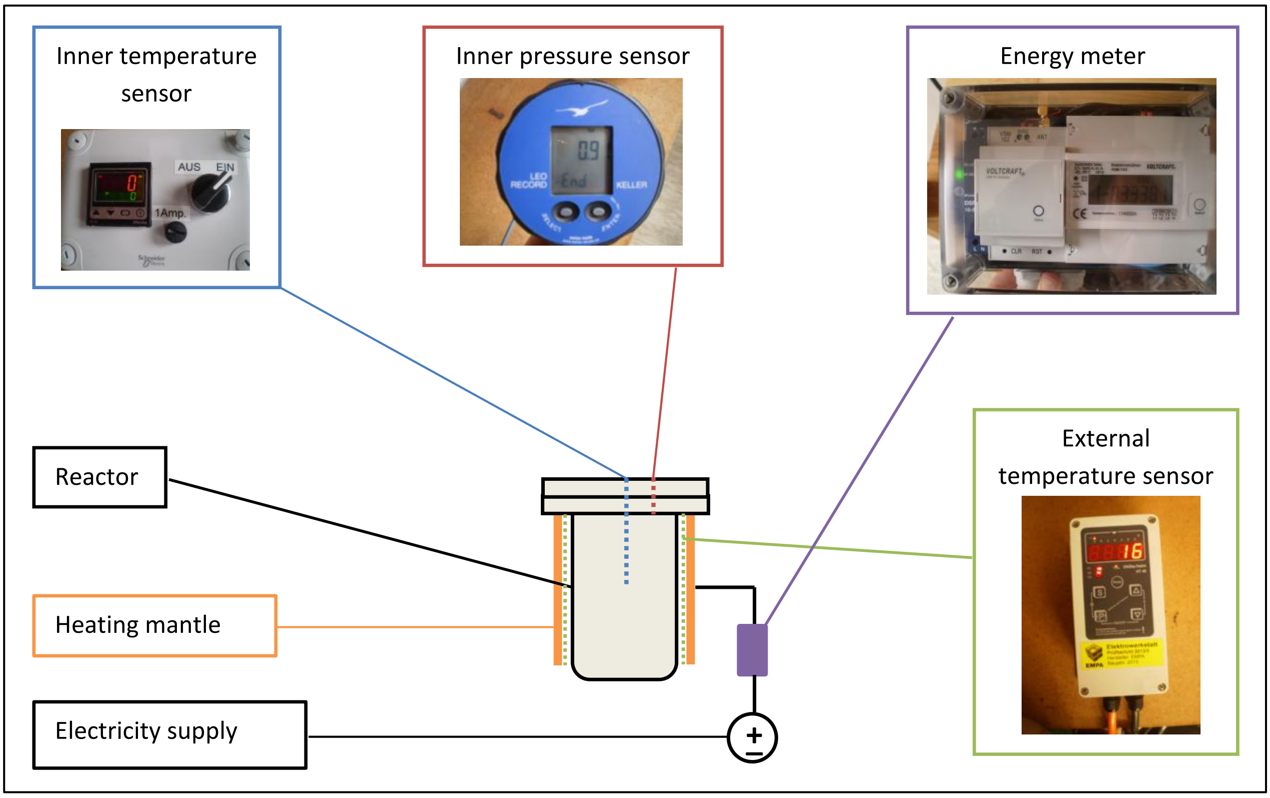


Figure 26: Experimental set up

6.1.2 Water test

For the water test, the reactor was filled with 17 Liters of water, closed and heated at different temperatures. The objective of the test is to get to know how the internal temperature and pressure reacts when the external temperature is increased progressively.

6.1.3 HTC tests

For the two experiments, first the rice was added in the reactor (1), then the water was filled up to 3/4 of the reactor's volume with water (2), corresponding to a filling volume of about 17 liters. The reactor was closed using a torque wrench at 84 Nm to tighten the screws (3).

Figure 27: Preparation procedure

The heat was applied during approximately 10 hours and then the reactor was let to cool down during the night. The next day, after releasing the remaining pressure (4), the reactor was opened (5), and emptied (6). The solid (7) was separated from the liquid (8) with a thick and tightly woven cotton cloth.

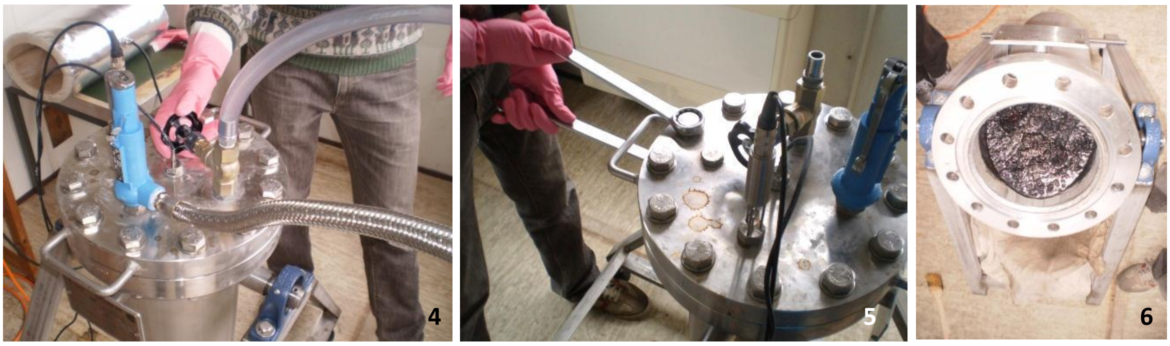
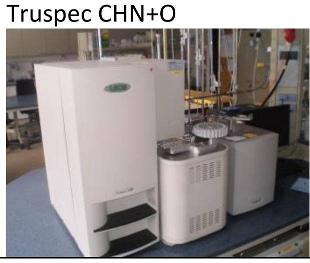


Figure 28: Emptying procedure

Figure 29: End products

6.1.4 Measurements

The following table summarizes the different measurements and methods used to analyze the output products (liquids and solids). One sample was taken from solids and liquids after each experiments. The total liquid and solids input and output were as well weighed before and after the reaction.

Table 27: Measurements and measurements methods				
	Measurement parameter	**Unit**	**Description**	**Measuring instrument or method**
Liquids	pH	-	Measure of the acidity of the process water after the reaction	Hach-Lange HQ D40
	EC	µS/cm	Measure of the electrical conductivity of the process water after the reaction	Hach-Lange HQ D40
	TOC	mg/l	Measure of the Total Organic Carbon present in the process water after the reaction	ZHAW: Photometer Lange DR3800, Test LCK 386 Eawag: Shimadzu TOC-L 720°C catalytic combustion
Solids	TS	%	Measure of the total solids of the HTC-coal and of the original substrate	ZHAW: Mettler Toledo HB 43-S Halogen heating Eawag: 24 hours in 105°C drying chamber
	Calorific value	J/g	Measure of the higher heating value of the dried HTC-coal and of the original substrate	Calorimeter IKA C200 
	Elementary analysis (C,H,O,N)	% by weight	Measure of the C, H, O and N contents of the dried HTC-coal and of the original substrate	Elemental analyzer LECO Truspec CHN+O 

6.1.5 Comparison with results from another reactor

These values are then compared with the values obtained with the HTC reactor Grenolmatik 25 (Grenol GmbH, Germany) at ZHAW. The reactor is composed of a double wall pressure vessel made of stainless steel with a detachable container of 25 Liters. It has a stirring device and an in-built heating mantle working with thermal oil. Four experiments were conducted with 1.13 kg of the same rice and 15.8 liters of water (total TS: 6.2 %): two with the use of stirrer and two without. The heat was applied during approximately 10 hours and the internal temperature was stabilized at around 205°C during minimum 4 hours. The comparison is carried out with the average of the results from the two experiments conducted without the use of stirrer. Results from these four experiments conducted at ZHAW can be found in the Appendix.

6.2 Results

6.2.1 Test with water

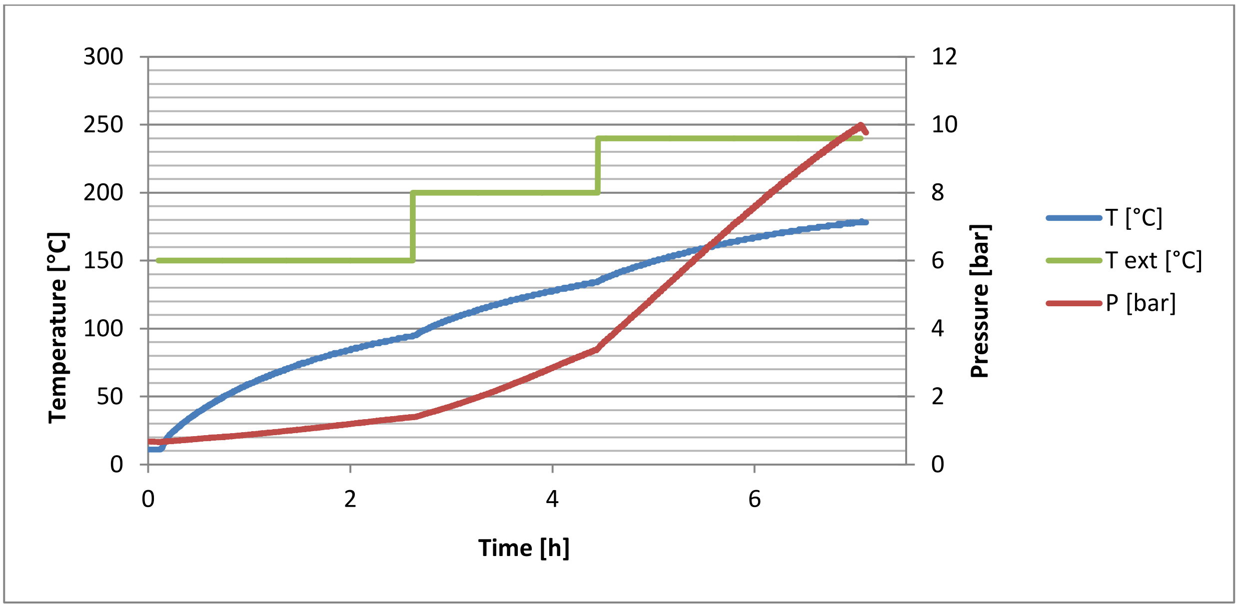


Figure 30: Graph of temperature and pressure for the test with water

The external temperature of the heating mantle was increased stepwise. The internal temperature rises slowly and reached 180°C after 7 hours. It was then decided to apply an external temperature of 240°C for the following experiment.

6.2.2 First HTC test

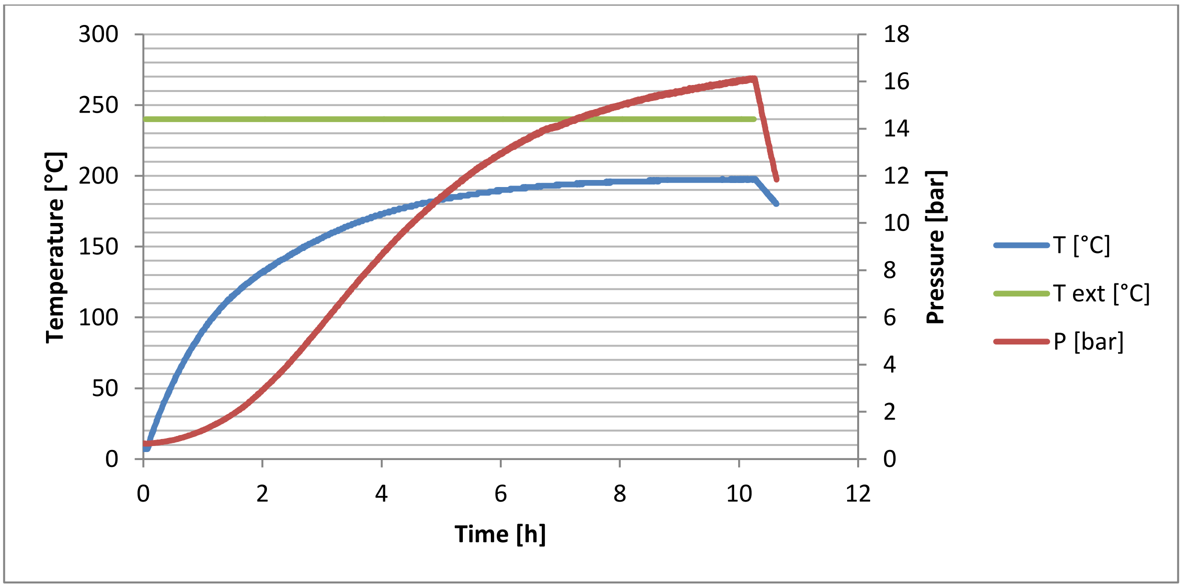


Figure 31: Graph of temperature and pressure for the first HTC test (TS of load: 2.6%)

The temperature stabilized around 200°C but never exceed it. The pressure reached 16 bars. As the experiments at ZHAW were conducted with an internal temperature of 205°C during minimum 4 hours, it was decided to use a higher external temperature for the next batch in order to reach comparable carbonization conditions.

Table 28: Energy consumption and duration of first HTC test			
Parameter	**Unit**	**Description**	**Value**
Energy consumption	kWh	Total electrical energy consumed by the heating mantle	12.4
Average power consumption	W	Average power consumed by the heating mantle	1263
Total reaction time	h	Time during which heat is supplied to the reactor	10.2
Reaction time	h	Total time with inner temperature above 180°C	6.0

6.2.3 Second HTC test

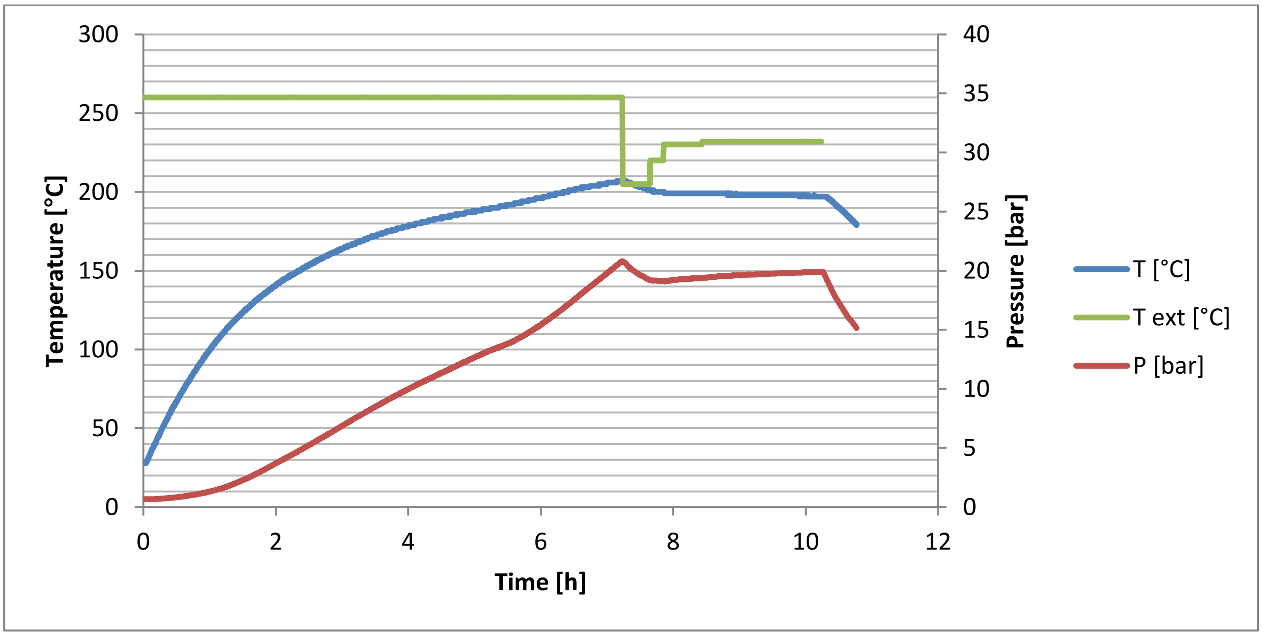


Figure 32: Graph of temperature and pressure for the second HTC test (TS of load: 5.3%)

The temperature increased to 207°C but didn't stabilize there, and the pressure went above 21 bar. Therefore the external temperature was decreased to 205°C after around 7 hours and then increased stepwise up to 232°C. The internal temperature then stabilized around 200°C during the remaining time (4.2 hours). With this second experiment, carbonization conditions comparable to the experiments conducted at ZHAW could be attained.

Table 29: Energy consumption and duration of second HTC test			
Parameter	**Unit**	**Description**	**Value**
Energy consumption	kWh	Total electrical energy consumed by the heating mantle	11.8
Average power consumption	W	Average power consumed by the heating mantle	1219
Total reaction time	h	Time during which heat is supplied to the reactor	10.2
Reaction time	h	Total time with inner temperature above 180°C	6.7

6.2.4 Outputs measurements and analysis

Table 30: Results of the measurements for HTC test 1 and 2, and comparison with results from ZHAW reactor					
		Original rice	**HTC-coal 1**	**HTC-coal 2**	HTC-coal ZHAW
In	Feedstock [kg]	-	**0.5**	**1**	1.1
	Water [L]	-	**16.8**	**16.6**	15.8
	TS [%]	88.2	**2.6**	**5.3**	6.2
Out	HTC-coal (wet) [kg]	-	**0.9**	**1.9**	2.3
	Process water [L]	-	**15.8**	**15.1**	15.1
Solids	TS output [%]	-	**10.8**	**18.1**	18.4
	HHV [MJ/kg dry basis]	17.7	**23.0**	**26.9**	27.7
	C [% dry basis]	44.1	**56.6**	**66.9**	69.4
	H [% dry basis]	6.5	**5.9**	**4.9**	5.2
	O [% dry basis]	49.4	**32.1**	**23.8**	22.7
	N [% dry basis]	1.2	**2.4**	**1.9**	2.2
Liquids	pH [-]	-	**3.6**	**2.7**	3.2
	EC [µS/cm]	-	**599**	**1083**	1038
	TOC [mg/L]	-	**4677**	**7764**	4933

With these measurement, the following parameters could be calculated:

Table 31: Results of analysis from measurements of the outputs					
Parameters		**Calculation**	**HTC-coal 1**	**HTC-coal 2**	**HTC-coal ZHAW**
Carbon mass balance	C in solids [%]	$\frac{\boldsymbol{C_{HTC-coal} * TS_{HTC-coal} * m_{HTC-coal}}}{\boldsymbol{C_{rice} * TS_{rice} * m_{rice}}}$	**26.9**	**58.4**	65.4
	C in liquids [%]	$\frac{\boldsymbol{TOC}\,[\mathrm{g}/\boldsymbol{l}] * \boldsymbol{V_{PW}}[\boldsymbol{l}]}{\boldsymbol{C_{rice} * TS_{rice} * m_{rice}}[\mathrm{g}]}$	**38.0**	**30.2**	18.8
	C in gases [%]	$\boldsymbol{1 - C_{solids} - C_{liquids}}$	**35.1**	**11.4**	15.8
Solid yield [% dry basis]		$\frac{\boldsymbol{TS_{HTC-coal} * m_{HTC-coal}}}{\boldsymbol{TS_{rice} * m_{rice}}}$	**21.0**	**38.5**	42.5
Energy content of HTC-coal [kWh]		$\frac{\boldsymbol{TS_{HTC-coal} * m_{HTC-coal}\,[g] * HHV\,[J/g]}}{\boldsymbol{3'600'000[J/kWh]}}$	**0.6**	**2.5**	3.2
Energy consumed [kWh]		**(measured)**	**12.4**	**11.8**	NA

6.3 Discussion

With the two HTC test, the heating value and the carbon content of the original substrate could be significantly increased, while the hydrogen and oxygen contents decreased. The heating value and carbon content of the HTC-coal from the first test are a bit lower than the ones from the experiments at ZHAW. This can be explained by looking at the severity of the reactions. During the first test, the inner temperature never exceeded 200°C whereas for the experiment at ZHAW, the temperature was stabilized at around 205°C during more than 4 hours.

For the second test, carbonization conditions similar to the one used at ZHAW could be reached. The measurements from the output products of both experiments give comparable results. In particular, the heating value and carbon content of both HTC-coals are relatively close to each other.

The first HTC test gives particularly low solid yield as well as C in solids. The second tests gives better results but values remain a bit lower than the ones from the experiments at ZHAW. Regarding the C in liquids, The big difference between the values from the two reactors can come from the fact that different methods were used to measure TOC in the process water (see table 27).

The coal produced in the second HTC test has a total energy content of around 2.5 kWh which is about one fifth of the energy required for the reaction (11.8 kWh). From this result it can be extrapolated that the HTC-coal produced from a rice input around 5 times bigger represents a comparable amount of energy than the amount needed for the reaction. This suggests that the energy requirements for HTC are relatively low.

7. RECOMMENDATIONS

7.1 REGARDING USE AND SAFETY

- When opening the reactor after HTC, protection mask, glasses, and gloves should be worn. Compounds like phenols, which have been measured and found to be present in the process water, vaporize and are corrosive to the eyes, the skin and the respiratory tract.
- When filling the reactor, before the start of HTC, it should be observed not to fill the reactor above three quarter of the reactor's volume (which means maximum 17 Liters). The water density decreases significantly at high temperatures resulting in a higher volume which can exceed the reactor's volume. See table 1 for the properties of water at high temperatures.
- Depending on the nature of the substrate, the resulting pressure during HTC can increase very fast. When using a new substrate for HTC, the reactor shouldn't be filled with a too high amount of feedstock (TS not above 5%). This allows for a better control of the pressure increase.
- The graphite sealing disc should be checked after every reaction. It should be replaced once in a while. If it's damaged it can be purchased from Aspag - Tel. +41 44 828 15 30 "Sigraflex-Flachdichtungen".
- The overpressure valve should be used as a safety device and not as a pressure regulation device. Therefore, it should be avoided to reach the maximal pressure of the overpressure valve (30 bar).
- In no way the drain valve should be opened during the reaction! This is used only to drain the residual pressure when the reactor is **cold** (below 40°C)**.**
- The imposed temperature on the heating mantle shouldn't exceed 300°C.
- The screws were closed with a torque wrench at 84 Nm and were numbered from 1 to 12. The tightening and opening of the screws shouldn't occur in the chronological order but should follow a cross pattern (example: 1-7-4-10-2-8-5-11-3-9-6-12). A first round with intermediate strength should be executed for example with 42 Nm before the screws are tightened with the maximum strength.

7.2 REGARDING POSSIBLE AMELIORATION

- One possible amelioration would be to connect the regulator of the heating mantle directly to the inner temperature sensor. This way, the regulation of the heat mantle occurs directly with the inner temperature of the reactor and not with the external temperature. This would allow to automatically control the temperature inside the reactor.

- Other than the implementation of a stirring device which, is a costly equipment, a possible amelioration would be to install a sampling valve, that can be used to extract samples of liquid and solid during the reaction and which can then be analyzed. This would help to better understand the HTC process. This system can be implemented with reasonable cost.
- During the reaction, the bottom of the reactor, which is not in contact with the heating mantle, gets cooled down by the ambient air. This sometimes results in an incomplete carbonization of the substrate that stays at the bottom of the reactor. A solution for that, other than the implementation of a stirrer, would be to paste some isolating material at the bottom of the reactor, to avoid the direct contact with the surrounding air.

7.3 REGARDING IMPLEMENTATION OF THE TECHNOLOGY IN DEVELOPING COUNTRIES

7.3.1 Reuse of waste heat

HTC is an energy intensive process - an issue when considering the implementation of the technology in developing countries. To minimize the energy consumption per unit of feedstock and to bring the technology to an economically viable application, the reactor needs to be operated in a multi-batch system where several batch HTC reactors are connected to each other and can be operated in parallel.

This way, the feedstock can be fed continuously in the system. Once the reaction is over in one reactor, the products are emptied and the HTC-coal separated from the liquid. The hot process water can then be redirected and reused directly to preheat the feedstock for the next batch.

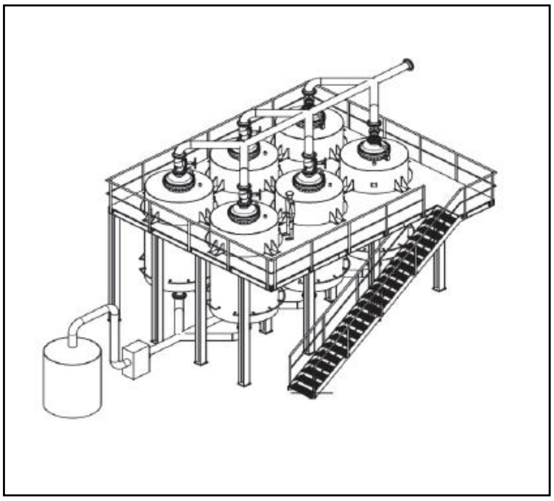


Figure 33: Schema of a multi-batch system (source: Krause, 2010)

7.3.2 Use of solar energy

Another way to avoid the dependency on the electricity grid is to make use of the solar energy available at the location. This way, the HTC reactor can be operated in a self-sufficient way in terms of energy consumption. The surface needed for a photovoltaic panel to power the reactor can be calculated from the energy consumption measured during the HTC tests. The energy needed for a reaction is 12.2 kWh per batch on average.

On the global insulation map, it can be noted that most developing countries are situated in the yellow region (yearly insulation of 1800-2000 kWh/m^2) which corresponds to a daily insulation of 4.9 - 5.5 kWh/m^2. On a bright day at earth's surface, the solar radiation reaches 1kW/m^2. This means that the daily insulation in a hot developing country can be estimated on average at 4.9 – 5.5 sun hours (for example 4.9 kWh/m^2 in Kumasi, Ghana; 5.2 in Madras, India; 5.5 in Lima, Peru)[3].

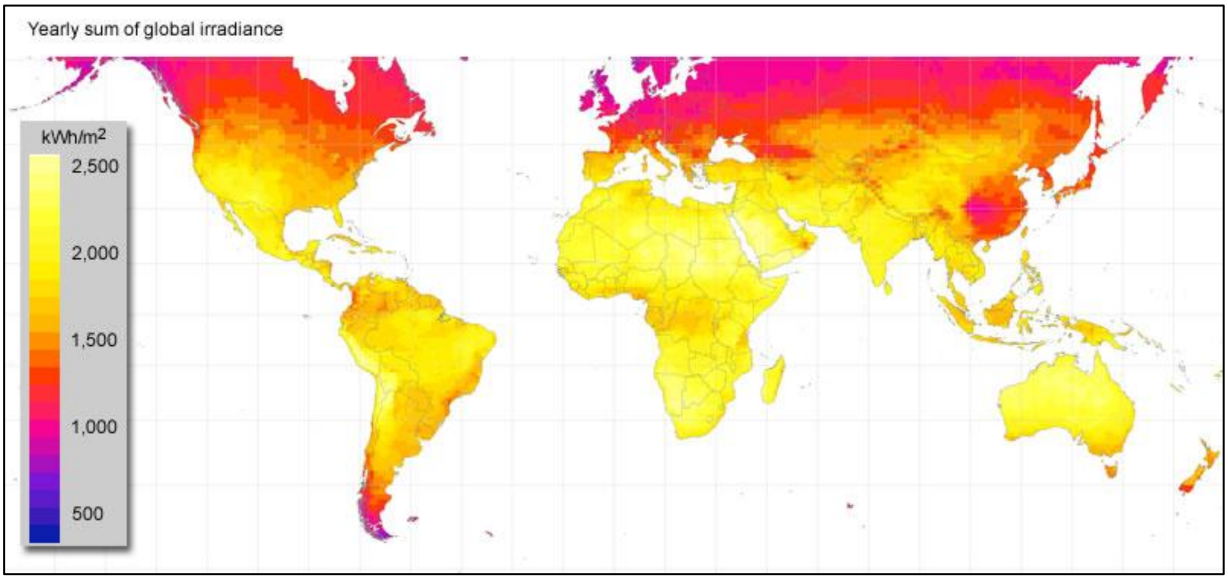


Figure 34: Global insulation map
Source: http://www.greenrhinoenergy.com/solar/radiation/empiricalevidence.php

Taking into account the energy losses and inefficiencies (factor 1.3), the wattage of the solar panel needed for the prototype reactor is

$$\frac{12.2\ \text{kWh}}{5.0\ \text{h}} * 1.3 = 3.2\ \text{kW}$$

For a wattage of 3.2 kW, a solar panel system with a surface area of about 22 m^2 is needed (efficiency around 15%). The possibility of using other systems that make use of the heat losses like PV/T cells should be investigated to increase the efficiency and thereby decreasing the surface area needed.

[3] Values taken from https://eosweb.larc.nasa.gov/sse/

Table 32: Summary of photovoltaic solar panel calculations	
Energy needed per batch	12.2 kWh
Daily sun hours	5
Wattage of solar panel needed	3.2 kW
Surface needed	22 m^2

7.3.3 Mixing substrates with different TS content

One requirement for the HTC is that the feedstock has to be completely submerged in water. In some cases, for example for dry substrates, this requirement might imply the addition of water for the process which can be an issue in some developing countries. In order to prevent this additional consumption of water, substrates of different moisture contents can be mixed together in order to obtain the required total moisture content. For example, liquid substrates like wastewater or digestate from biogas plants can be mixed with substrate containing less moisture like biowaste or fecal sludge. This way the feedstock can attain the required moisture content without addition of water.

8. CONCLUSION

In comparison to other HTC reactors of a similar volume, the developed HTC prototype reactor is a simple system at low cost and complexity. Its design has been selected according to suitability criteria for application in developing countries and such that it can be operated in a safe and easy way. HTC tests carried out with a model substrate show that the required reaction conditions can be reached and that HTC-coal with comparable characteristics than the one produced with a state of the art reactor can be produced. Thus the reactor can be used to assess the suitability of the technology for treatment of organic waste in developing countries.

HTC is a process that has a multitude of interesting aspects and offers lots of opportunities, particularly for developing countries. It can be used to treat problematic waste like fecal sludge, while producing a hygienic and valuable product with relatively low energy requirements. Furthermore, HTC-coal can be used as soil conditioner or a carbon neutral energy carrier, eventually substituting traditional fuels like fossil coal or firewood and contributing to avoid deforestation.

However, this system requires high temperatures and high pressures to function, which implies some rigorous requirements with respect to the materials as well as construction procedure. The design and dimensions as well as the type of materials for the reactor have to be determined according to strict conformity standards and in agreement with the applicable regulations. These require all the materials, design, calculations, construction procedure (as welding) to be made by qualified personal and to be supervised and certified by an entitled authority.

The energy requirements, the treatment of the process water as well as the need for water depending on the type of feedstock remain problematic aspects of the technology in developing countries. The reuse of waste heat, the use of solar energy and the possibility to mix substrates with different water contents are solutions to be further investigated to tackle these issues.

Table 33: SWOT analysis	
Strengths	**Weaknesses**
Simple and low tech system Low construction costs compared to other similar reactors Easy and safe operation Produces valuable coal from waste biomass, with relatively low energy requirements Provide hygienic and efficient treatment of problematic waste like fecal sludge	Process requires high T and P Construction implies rigorous design and construction requirements Process requires energy Requires the use of water if feedstock not sufficiently wet Large amount of process-water produced
Opportunities	**Threats**
Substitute firewood in developing countries (deforestation) Reduces GHG emission if substituting fossil fuels like coal Improvements of soils if HTC-coal used as soil amendment Sequestration of CO2 if the coal is applied to the soil Reduction of waste burden and improvement of health and environmental situation in developing countries	Post-treatment of process-water remains a problematic issue The necessary materials (stainless steel), infrastructures (pressure vessel engineering), or qualified personal (welders) to build a HTC reactor might not be available in all developing countries Requirements in terms of energy supply might be an issue in developing countries

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APPENDIX

a) RESULTS FROM EXPERIMENTS AT ZHAW (WÄDENSWIL)

Four experiments were carried out using rice. Two with the use of a stirrer during the reaction (MR1 and MR2) and two without (OR1 and OR2).

i) Analysis of outputs

Reis vor HTC

TS [%]	88.21
	88.09
TS Mittelwert [%]	88.15
Brennwert [J/g]	17726
	17609
Brennwert Mittelwert [J/g]	17668
Ctot [%]	44.08
	44.03
	44.12
Ctot Mittlewert [%]	44.08
H [%]	6.49
	6.48
	6.46
H Mittelwert [%]	6.48
O [%]	49.22
	49.51
O Mittelwert [%]	49.37
N [%]	1.170
	1.013
	1.370
N Mittelwert [%]	1.184

HTC von Reis

	MR1	MR2	OR1	OR2
Dauer [h]	10	10	10	10
T [°C]	205	205	205	205
P [bar]	25	25	25	25
Enddruck [bar]	1.8	2.6	3.6	3.8
Endtemperatur [°C]	38.5	40.6	48.1	49.3
Menge vor HTC				
Behälter leer [kg]	7.809	7.823	7.8155	7.8135
Biomasse vor HTC [kg]	1.1345	1.1335	1.1365	1.137
Wasser vor HTC [kg]	15.788	15.788	15.79	15.78
Füllstand des Behälters vor HTC [cm]	18.5	18.8	18.3	18.6
Wasser zugegeben aussen [kg]	1.7085	1.554	1.5655	1.5525
Menge nach HTC				
Füllstand des Behälters nach HTC [cm]	18.3	18	15.5	15.4
Behälter + Kohle + Prozesswasser nach HTC [kg]	24.7775	24.643	25.795	25.752
Behälter leer nach HTC [kg]	8.0065	7.94	7.9655	7.9795
Kohle nach HTC [kg]	2.0744	1.9535	2.0795	2.549
Wasser zugegeben aussen nach HTC [kg]	0.1125	0.086	1.168	1.113

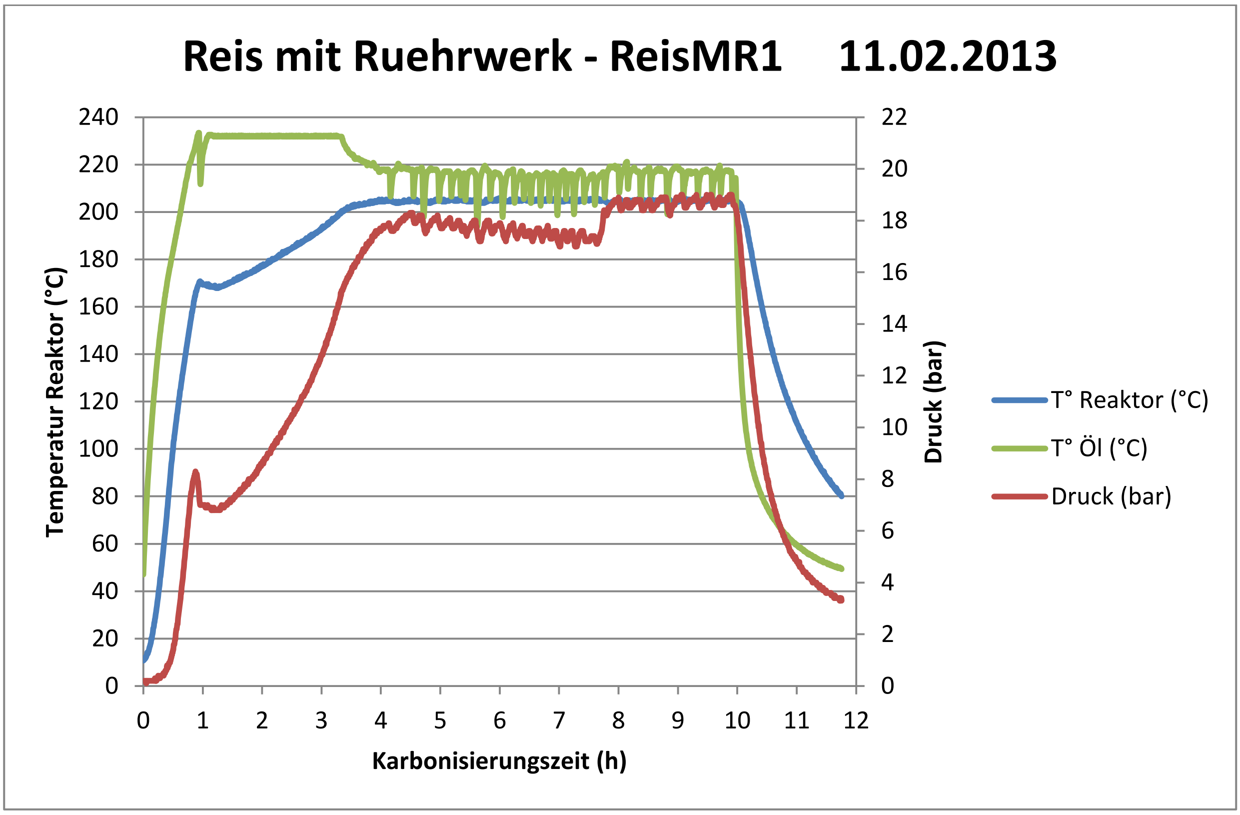
Prozesswasser				
Menge	14.4915	14.454	15.3075	14.957
pH	2.93	3.2	3.22	3.07
	2.95	3.21	3.17	3.12
	3.05	3.21	3.23	3.15
pH Mittelwerte	2.98	3.21	3.21	3.11
EC [µS/cm]	1177	1117	1057	1012
	1170	1121	1054	1026
	1163	1126	1053	1028
EC Mittelwerte [µS/cm]	1170	1121	1055	1022

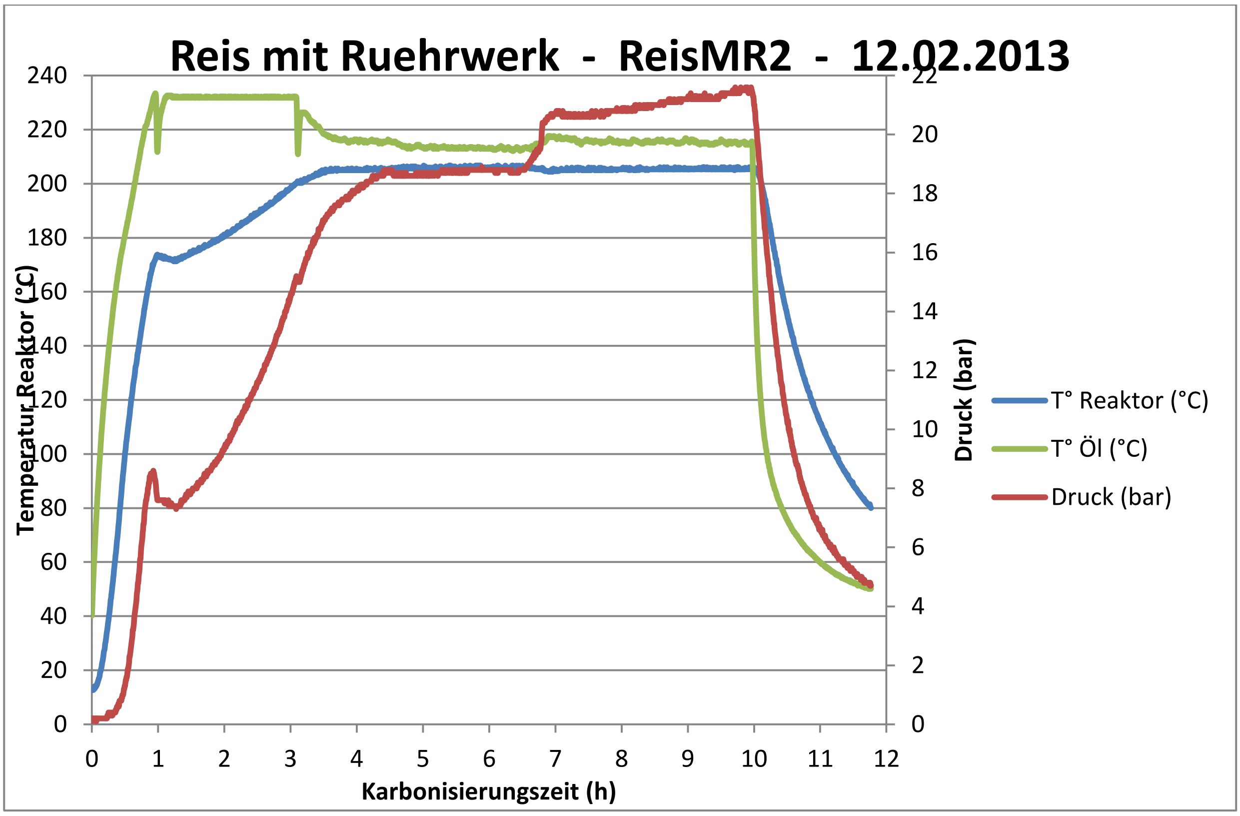
phenole [mg/l]				
Vorversuche mit verschiedenen Verdünnungen				
1:50	6.07			
1:100	3.39			
1:500	0.751			
1:1000	0.369			
1:2000	0.18			
phenole [mg/l]	1.55	1.61	1.61	1.56
1:200	1.64	1.54	1.56	1.45
	1.68	1.56	1.57	1.56
	1.63	1.56	1.58	1.52
	1.64			
phenole Mittelwerte [mg/l] 1:200	1.63	1.57	1.58	1.52
phenole Mittelwerte [mg/l]	325.60	313.50	316.00	304.50
TOC [mg/l]				
Vorvesuche mit verschiedenen Verdünnungen				
1:50	124			
1:100	63.5			
1:500	26.4			
1:1000	9.91			
1:2000	6.29			
TOC [mg/l]	55.1	51.8	50.8	48.1
1:100	58	57.1	48.8	49.3
	52.6	49.7	51.4	47.6
	60.4	50.9	49.4	49.2
TOC Mittelwerte [mg/l] 1:100	56.5	52.4	50.1	48.6
TOC Mittelwerte [mg/l]	5652.5	5237.5	5010.0	4855.0

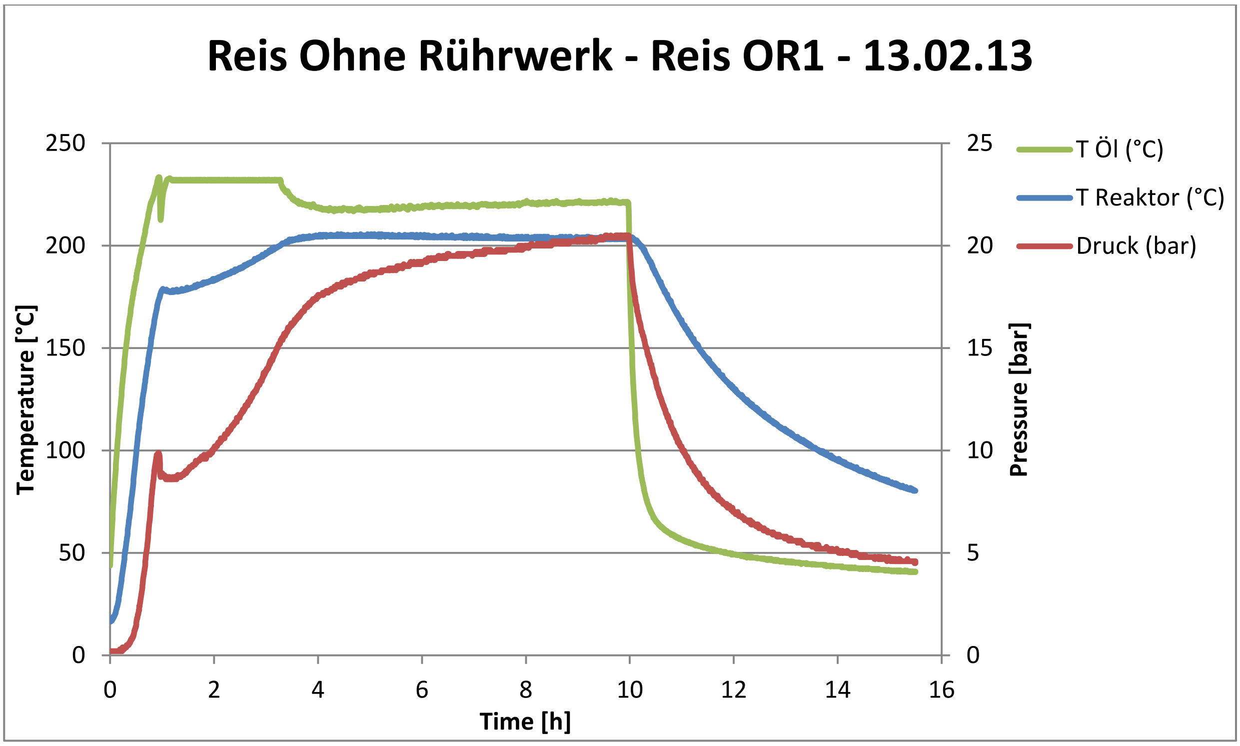
Kohle

TS [%]	20.13	21.34	19.19	16.46
	18.87	20.81	21.54	16.02
	17.86	21.61	20.79	16.36
TS Mittelwerte [%]	18.95	21.25	20.51	16.28
Brennwert [J/g]	27810	27332	27397	27843
	28011	27336	27448	27949
Brennwert Mittelwerte [J/g]	27911	27334	27423	27896
Ctot [%]	70.25	69.00	69.25	69.76
	70.51	68.83	69.08	69.48
Ctot Mittelwerte [%]	70.38	68.92	69.17	69.62
H [%]	5.00	4.97	5.18	5.15
	5.02	4.96	5.17	5.14
H Mittelwerte [%]	5.01	4.97	5.18	5.15
O [%]	23.32	23.00	22.65	22.54
	23.51	22.47	22.90	22.51
O Mittelwerte [%]	23.42	22.74	22.78	22.53
N [%]	2.190	2.264	2.163	2.208
	2.215	2.267	2.317	2.198
N Mittelwerte [%]	2.203	2.266	2.240	2.203

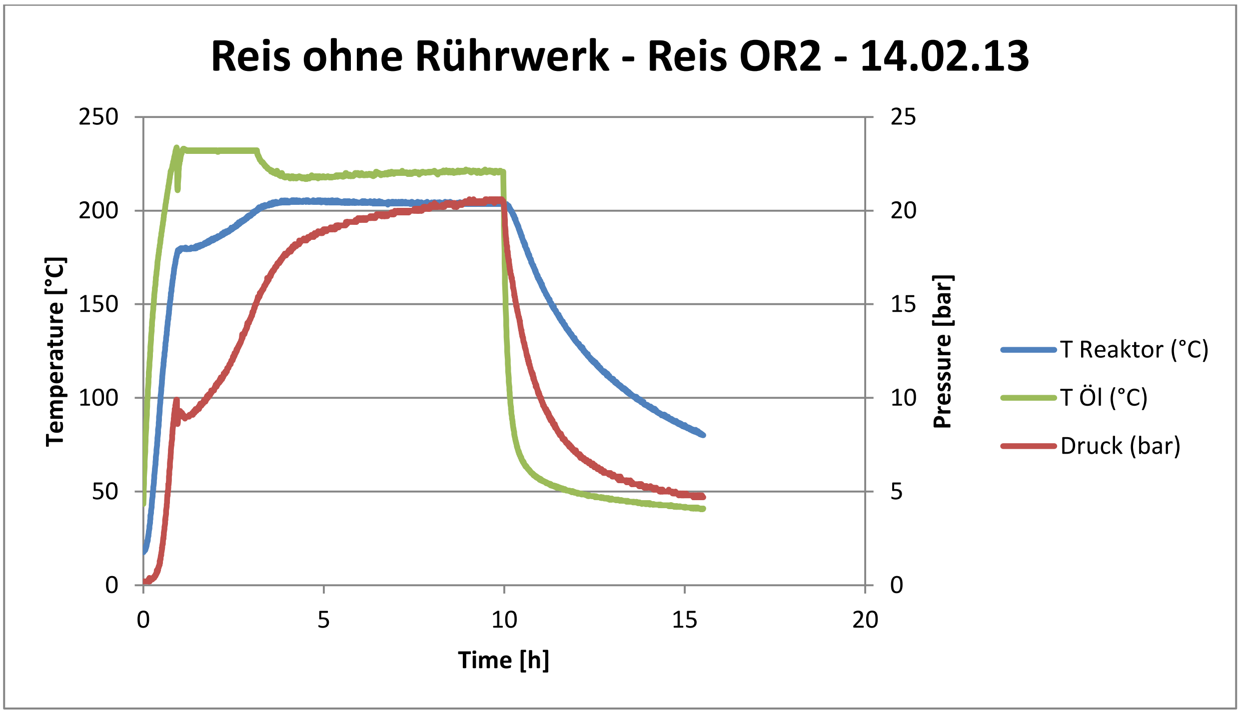
ii) **Graph of pressure and temperature**







Reis Ohne Rührwerk - Reis OR1 - 13.02.13
250
200
150
100
50
0
Temperature [°C]
0
2
4
6
8
10
12
14
16
Time [h]
25
20
15
10
5
0
Pressure [bar]
T Öl (°C)
T Reaktor (°C)
Druck (bar)



Reis ohne Rührwerk - Reis OR2 - 14.02.13
250
200
150
100
50
0
Temperature [°C]
0
5
10
15
20
Time [h]
25
20
15
10
5
0
Pressure [bar]
T Reaktor (°C)
T Öl (°C)
Druck (bar)

b) Magnetic coupled stirrers

These are example of magnetic coupled stirrer from the following company:

ALOWAG AG Pumpen Rührwerke / Pompes Agitateurs

Duggingerstrasse 2

CH-4153 Reinach-Basel

Tel : + 41 061 711 66 36

Direkt : + 41 061 715 96 34

Mobile : + 41 079 646 28 81

Fax : + 41 061 711 68 06

e-mail : T.Lehmann@alowag.ch

Internet : www.alowag.ch

Magnetrührwerke für die Hydrothermale Carbonisierung (HTC)

Magnetrührwerk M-10

Magnetrührwerk M-20

Magnetrührwerk in Ex-Ausführung