Hydrothermal Liquefaction of Wet Waste Streams

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Summary

Wet waste streams include a wide variety of products such as food processing residues, sewage sludge but also the organic fraction of municipal solid waste. Humidity typically varies from 50 to 90%. These waste streams are often used or abandoned in low value applications such as composting, incineration or landfill. Many environmental problems are associated to the waste streams such as bad odours but also due to the production of secondary pollutants such as dioxins during incineration. Basic incineration but also more advanced techniques such as gasification and pyrolysis are interesting for dry feedstocks but lose much of their interest when the humidity of the resource is higher than 50%. Dewatering and drying is possible for most feedstocks but at a significant cost. Hydrothermal liquefaction produces a biocrude that can be further upgraded to biofuels. Hydrothermal conversion takes place in the water phase and produces a hydrophobic product making separation of the final product easier. This paper gives some examples of how hydrothermal liquefaction can produce a biocrude or a heavy fuel oil. The production costs are high compared to their fossil counterparts but gate fees in the order of 50-130 € ton^{-1} ensure economic competitiveness.

Introduction

Wet waste streams are a relatively abundant low value waste stream and exist in a wide variety of forms including sewage sludge, food processing residues, the wet fraction of municipal solid waste, and others. These wastes have a low energy content making incineration costly. Composting and anaerobic digestion are often proposed for these waste types. This paper shows how Hydrothermal Liquefaction can (HTL) increase the value of wet waste by transforming it into an fossil fuel replacement/

Hydrothermal liquefaction converts biomass in hot compressed water into a biocrude. This biocrude is an oily material containing bio-oil and char. Hydrothermal liquefaction has been known for some time. The developments started simultaneously in Europe [1] and in the United States [2]. The conversion takes place at temperatures between 300 and 400°C and at pressures above the saturation pressure to ensure that water remains in the liquid phase, typically above 100 bar. Under these conditions the ionisation of water increases while its polarity decreases, favouring depolymerisation and dehydration of biomass polymers to produce hydrophobic compounds.

Figure 1 shows a typical resource, blackcurrant pomace, an autoclave reactor and the biocrude obtained. The Higher Heating Value of the biocrude is typically 30-35 MJ/kg. The biocrude can either be used directly as a combustible, fed into a refinery as crude oil [3], or it can be upgraded to a diesel type biofuel [4]. The development of the technology has been hampered by low oil prices but also by technical difficulties and the increasing cost of biomass. The application of hydrothermal liquefaction to wet waste streams can procure a new momentum for this technology. Traditional HTL laboratories such as PNNL are actively working on this subject as well as many newcomers.

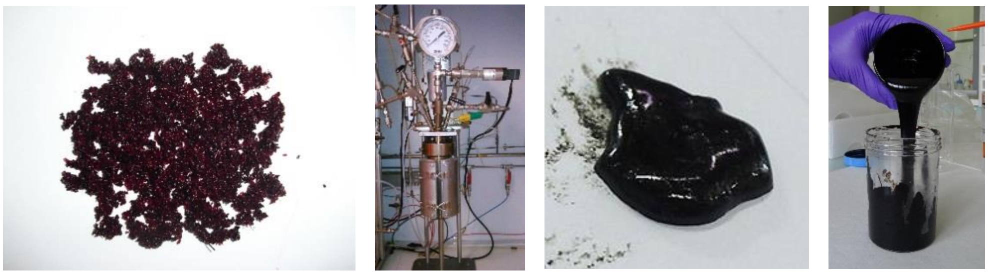

Figure 1: Example of the resource blackcurrant pomace, the HTL batch reactor and the obtained biocrude.

The chemical composition of the resource plays a major role in the product yield and quality as shown by Déniel at al. [5]. Important parameters include ash content, fibre composition and content, proteins and lipids. This study looks at biomasses rich in lipids and proteins and also a purely ligno-cellulosic biomass. It has been shown that certain additives [6] and operating conditions [7] also greatly influence biocrude and bio-oil yields but also their quality.

There are many technical-economic evaluations of biomass to fuel processes. There are however few evaluations of HTL processes, most on the conversions of biomass into biofuel. The majority of the evaluations of the HTL process are done on either algae [8,9] or wood [1,4]. Other studies exist on swine manure as a resource [3,10]. Typical production costs for diesel type fuels from cultivated algae are in the 2-3 € L^{-1} range [8] considering a fully integrated production site. Prices of defatted (waste) algae are much lower as the extracted lipids are sold at a premium price. HTL fuels from defatted algae may be much cheaper, less than 1 € L^{-1} [9] for very large plants (2000 ton day^{-1}). Wood conversion plants at a large scale are also expected to be (nearly) profitable at a large scale with production prices in the 0.6-1.2 € L^{-1} [4,1] range. More complicated feedstocks such as sewage sludge and swine manure received less attention for technical-economic evaluations. Buisonjé [3] estimated that an integrated swine manure conversion plant should be economically viable with a gate fee of at least 15 € ton^{-1} to produce a biocrude that can be sold to a refinery for further upgrading.

Sewage sludge conversion in HTL plants has an additional challenge in that the resource is very distributed and that transport of wet sludge over significant distances is not recommendable. Local processing should be favoured. A typical metropolitan area as Grenoble (France) produces around 7000 ton of dry sewage sludge per year, around 1 ton dry matter per hour, or around 10 m^3 per hour of biomass slurry. The optimal residence time in the reactor will probably range from 10-20 minutes depending on the resource and the temperature. This means that the reactor volume should be around 2.5 m^3 which is already quite large for a pressurised reactor. Being limited to low scales makes the economic viability even more difficult. Gate fees are common place in the waste treatment industry and typically 100-200 € ton^{-1} is charged for waste treatment in France [11]. The use of sewage sludge as an agricultural resource is more and more constrained and is also costly [12], in the same orders of magnitude.

The focus of this paper is on wet solid wastes such as food processing wastes and municipal sewage sludge. Many other resources are suitable for hydrothermal liquefaction, such as micro and macro algae or even dry resources such as wood. The actual resources presented in this study include grape marc and blackcurrant pomace representing food processing residues. Three types of sewage sludge are also tested, activated, secondary, and digested sewage sludges. These resources are characterised by a humidity varying from 50- 90 wt. % and an extremely variable chemical composition. The analysis of the resources by following regular food analysis norms for fibres, lipids and proteins.

Hydrothermal liquefaction produces a biocrude with an interesting energy content [3]. The actual market value of the product is unknown. The biocrude can be further separated into bio-oil and char by means of solvent extraction. The produced oil can be compared to heavy fuel oil [13]. This bio-oil can be further refined into a biofuel by catalytic upgrading, typically a biodiesel [4]. The higher the degree of refinement considered, the more uncertain the technical and economic feasibility is.

The objective of this study is to show how these low value resources can be upgraded to biofuels. The paper presents experimental results of how different resources behave under hydrothermal liquefaction conditions. The emphasis of this paper is not on the experimental work. The product yields of different resources, converted at the same conditions, are used to estimate the cost of the hydrothermal conversion. Gate fees are estimated to ensure economic viability of the plants.

Materials and Methods

The food processing residues presented in this study include are grape marc and blackcurrant pomace representing food processing residues. These are procured via local producers (UNGDA and Les Vergers Boiron). Three types of sewage sludge were tested, activated, secondary, and digested sewage sludge from municipal sewage treatment plants in the Grenoble region in France (Aquantis in Voreppe and Aquapole in Le Fontanil).

The resources have been analysed by well-known techniques to establish the chemical composition of the resource. The results are presented in Table 1. Simple sugars cannot be quantified by standard methods and are typically calculated by difference (everything that is not ash, protein, lipid or fibre).

Table 1: Characterisation of blackcurrant pomace, grape marc and sewage sludge used in this work

	Blackcurrant pomace	**Grape marc (dried)**	**Sludge 1 Mixed**	**Sludge 2 Biological/ Activated**	**Sludge 3 Digested**
Moisture content (wt. %) [1]	59.6	7.4	83	94	97
Fibre content (wt. % of dry matter) [2]	62	70	40	38	50
NDF (*Neutral Detergent Fibres*)	62	70	40	38	50
ADF (*Acid Detergent Fibres*)	53	63	28	30	26
ADL (*Acid Detergent Lignin*)	35	49	21	7	18
Cellulose (*ADF-ADL*)	18	15	7	23	8
Hemicelluloses (*NDF-ADF*)	9	6	12	8	25
Lignin (*ADL*)	35	48	21	7	18
Proteins (wt. % of dry matter) [3]	17	9.7	11	5	3
Lipids (wt. % of dry matter) [4]	15	8.1	10	15	13
Ash content at 550°C (wt.% of dry matter) [5]	4.3	4.8	14	14	38

[1] EN 14774-1 [14].
[2] NF V18-122 [15]
[3] Kjeldahl method
[4] Hydrochloric acid digestion + Petroleum ether extraction
[5] NF EN 14775 [16]

Batch experiments were performed in a 0.6 L stainless steel (SS316) stirred batch reactor (Parr Instruments Company). In a typical experiment, the reactor is filled with approximately 240 g of biomass slurry, with a constant 14 wt. % dry matter to water ratio. In certain cases, in particular for sewage sludge, a slurry with 14 wt. % dry matter is too viscous and the mixture is further diluted to 10 wt. % dry matter. The autoclave is leak tested, purged and pressurized to 1 MPa with nitrogen gas, to ensure sufficient pressure for gas analysis after the reaction. The pressure inside the reactor is a function of the reaction temperature, the amount of water and the amount of produced gas during the process. The reactor is stirred at 600 rpm and is heated to the reaction temperature by an electrical heater. Once the reactor reaches the reaction temperature, it is held during a specified time within ± 1°C of the specified operating temperature (holding time). For these experiments a 15 min holding time is applied. All resources have been treated at 300 °C, this temperature is reached in about 35 minutes. After the holding time, the reactor is rapidly cooled to room temperature in 20 min by an air quench.

After venting the reactor for gas analysis, the content of the reactor is first filtered on a Buchner filter to separate the aqueous phase from the raw organic residue. The raw organic residue is sticky, and removed from the reactor as best as possible. The reactor is then weighed and the weight difference with the empty reactor is counted as raw organic residue. The produced biocrude, the raw organic residue, is dried at room temperature under air circulation until a stable mass was obtained. The procedure is further detailed in the Figure 2.

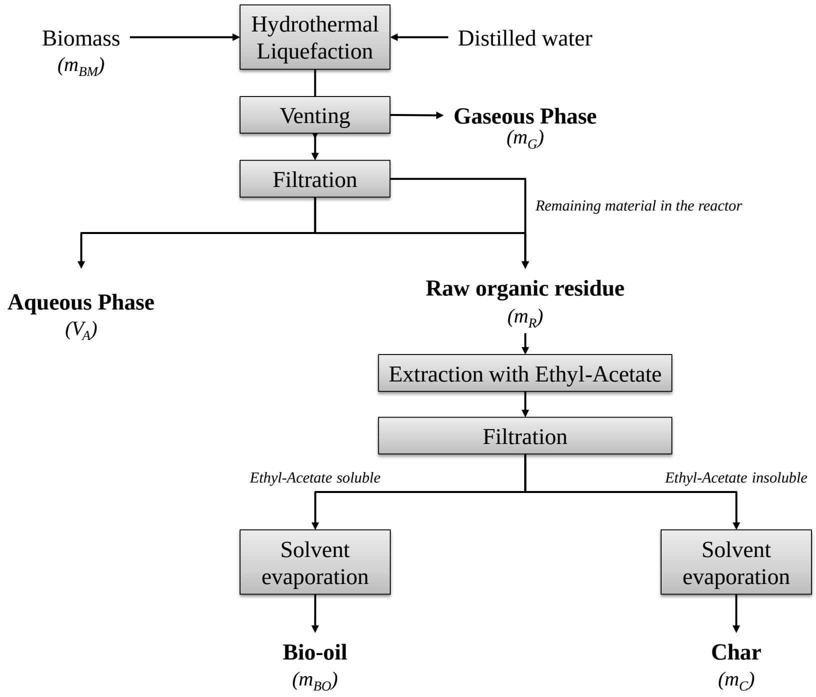

Figure 2: Products recovery procedure after hydrothermal liquefaction

The biocrude is separated into char and bio-oil using a solvent, ethyl-acetate in our case. Bio-oil is recovered after evaporation of the solvent at room temperature under air circulation, until a stable weight is obtained. GC-MS analysis confirmed that no residual solvent is left in the bio-oil. The char is also dried at room temperature under air circulation, until a stable weight is obtained. Weight loss of the char after extraction and drying is used to determine the proportion of solvent-soluble organics in the raw residue, and therefore the bio-oil yield. All yields reported in this study are expressed in weight percentage of the dry biomass (wt. % dry matter).

The technical-economic evaluation is based on a process simulation with the ProSimPlus software [17]. The simulation was used to calculate equipment sizing (heat exchanger surfaces, heating requirements, etc.). The evaluation of the equipment cost and economic evaluation is based on the methods describes by Turton [18] and Chauvel [19]. The main economic parameters as they enter in the production costs are presented in Table 2. The total installed equipment cost (Inside Battery Limits, ISBL) serves as a basis to estimate the overall investment (CAPEX), including buildings, utilities and the engineering. The approach is that the plant is located on an existing industrial site, either a food processing plant or a waste water treatment plant (WWTP). Minimal additional personnel costs are therefore necessary.

Table 2 : Financial parameters for the economic evaluation

Discount rate	8 %
Part bank loan in investment	50 %
Stream factor	7000 h $year^{-1}$
Capital depreciation	10 years
Loan duration	10 years
Technical lifetime	40 years
Tax rate	30 %
Personnel	5 Full Time Employees (FTE)
Personnel costs FTE	70 k€ $year^{-1}$ FTE^{-1}
Electricity price	150 € MWh^{-1}
Treatment cost waste water	0.5 € m^{-3}
Salvage value plant	10 % du CAPEX

Discounted cash flow methods take into account the variation of the value of the invested money and the value of the revenues. Discounted methods discount operating costs and revenues in time. A euro earned in

2017 does not have the same value to a company as a euro in 2027. The cash flow (*CF*) in any operating year *n* is discounted to a "present value".

$$CF_n = \frac{Revenues^{Year-n} - Costs^{Year-n}}{(1+i)^n}$$
Eq 1

The sum of the discounted investment, all yearly cash flows and the salvage value of the plant is the Net Present Value (*NPV*) of the project after *N* years.

$$NPV = DeprCapCost + \sum_{n=1}^{N} CF_n + \frac{Salvage}{(1+i)^N}$$
Eq 2

The minimum selling price is found by imposing the *NPV* to zero with a selected depreciation time. This method generally yields a higher production cost than the simple production cost as presented earlier. It also allows us to take into account depreciation, inflation, taxes and the salvation value of the plant.

Experimental Results

Batch experiments always produce a mixture of solids (char), extractable (bio-oil) and an aqueous phase rich in ash and organic molecules. The products are separated according to the procedure describes earlier. The results of the experiments are presented in Table 3.

Table 3: Results of batch liquefaction experiments at 300°C

Yields	**Blackcurrant pomace**	**Grape marc**	**Sludge 1 Mixed**	**Sludge 2 Biological/ Activated**	**Sludge 3 Digested**
Biocrude (%)	52	35	51	61	54
HHV Biocrude (MJ kg^{-1})	32	*30*	26	24	13
Char (wt. %)	27	22	27	35	37
Bio-oil (wt. %)	25	13	24	26	17
Gas (wt. %)	12	8.0	5.5	8.5	6.6
Aqueous phase (by difference) (wt. %)	24	57	44	31	40

We observe significant variations between the results from the different resources. Resources rich in lipids and proteins such as some sewage sludge but also blackcurrant pomace produce significant amounts of oil. The lipids initially present in the resource clearly help increasing the bio-oil yield. It is clear that lignin rich resources such as grape marc produce less oil than other resources under these conditions. Digested sewage sludge is very rich in ash and as a consequence contains less organic material. In addition, the organic material remaining after anaerobic digestion contains few proteins and lipids. It has lost much of its proteins and lipid content, making it less interesting for HTL.

Some of the sulphur is found in the gas phase as hydrogen sulphide. The gas produced by HTL contains mainly CO_2 and is generally badly smelling. This gas needs to be oxidised in the combustion furnace before it can be vented to the atmosphere. . The aqueous phase contains a significant amount of organics and cannot be disposed without further treatment. The process water is treated in a water treatment plant.

Technical-Economic Evaluation

The technical-economic analysis is presented on these five cases. Two different cases are presented. A simple conversion plant that produces biocrude that is sold to a refinery as a crude oil replacement. The same conversion plant is equipped with a solvent extraction unit to produce a bio-oil. Bio-oils are acid corrosive [20,13], this means that stainless steel should be used as construction material.

The biocrude plant is described in Figure 3. The plant consists of a feed preparation and heating. Most of the required heat is recovered from the product stream. The products are sticky when cold. Full heat recovery is therefore problematic as heat exchangers tend to foul when the biocrude contacts cold surfaces. Some additional heating is therefore necessary. The tubular reactor converts the feed into a biocrude. The residence time is 15 minutes. The products are cooled by heating the feed and the aqueous phase, the biocrude and the gas are separated. The water is recycled to the waste water treatment works. Globally, the amount of water is not very large. In the case of sewage sludge, the process water is locally reprocessed. In the case of the blackcurrant pomace and grape marc, process water is sent to external water treatment plant leading to additional costs in these cases.

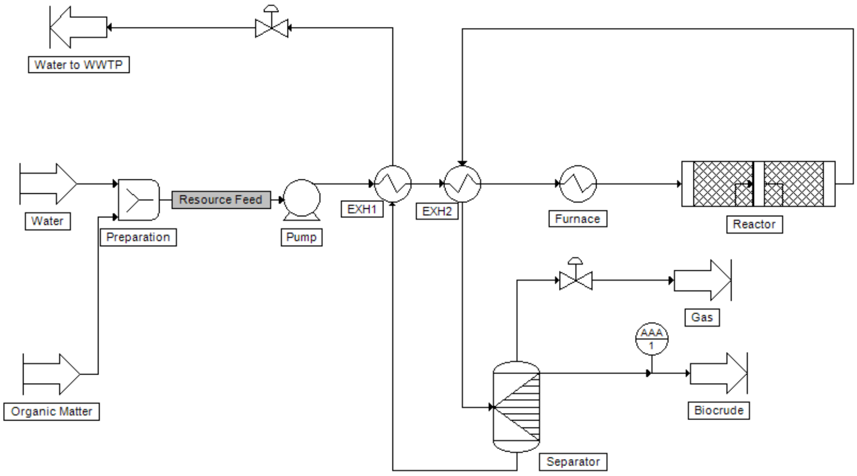

Figure 3: Process scheme biocrude plant

The water content affects the results of the liquefaction, it has been shown that increasing dry matter content decreases the oil yields of the process [7,21]. Increasing the water content also increases the volume of the installations. The water content in the feed is 90 wt. % (10 wt. % dry matter) in this study. This ensures good pumpability and optimal yields. The sewage sludge can easily be dewatered to the desired water content. Grape marc and blackcurrant pomace will have to be diluted with process water. This actually has a beneficial effect on the yield and the biocrude quality [22].

The different cases are evaluated and presented below in Table 4. In practice, as the volumes of slurry are the same, the investment costs are very similar. Fixed and operating costs are also very close between the different cases. The differences are in the yields and the energy content of the products. The gate fee is calculated in the cases when the biocrude production costs are higher than the reference crude oil price. The gate fee is the negative value of the feed to make sure the products can be sold without further losses. When the gate fee is lower that alternative disposal ways, the operation is beneficial.

Table 4: Economic evaluation of a biocrude plant

	Blackcurrant pomace	Grape Marc	Sludge 1	Sludge 2	Sludge 3
Investment (CAPEX) – M€	2.70	2.70	2.70	2.70	2.70
Fixed costs – M€ year^{-1}	0.46	0.46	0.46	0.46	0.46
Variable costs – M€ year^{-1}	0.78	0.78	0.78	0.78	0.78
Minimum selling price – € ton^{-1}	333	556	584	623	980
Minimum selling price – € GJ^{-1}	10	19	22	26	76
Gate fee – € ton^{-1} dry matter	50	95	100	105	132
Crude oil (Brent 2015) – € GJ^{-1}	6.7				
Fossil coal (2015) – € GJ^{-1}	1.5				

The second case concerns the same plant extended with a solvent extraction unit to separate the biocrude into bio-oil and char as shown in Figure 4. A solvent is mixed into the biocrude stream. Char is separated from the mixture as the insoluble part. The solvent is separated from the bio-oil by distillation. The biocrude contains insoluble char, heavy oil but also light compounds [20,13]. In practice, the initial solvent will be rapidly replaced by the light compounds included in the biocrude that are separable by distillation.

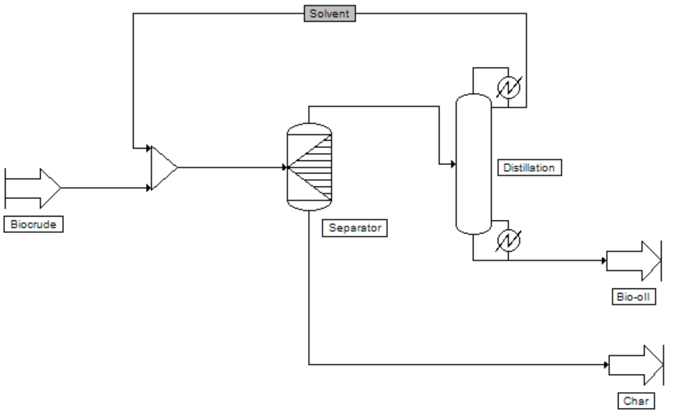

Figure 4: Process scheme solvent extraction unit

The different cases are evaluated and presented below in Table 5. The investment costs are now dependant on the amount of oil produced. The production costs only concern the oil produced, the char is used as fuel for the process and is not further valorised.

Table 5: Economic evaluation of a fuel oil plant

	Blackcurrant pomace	Grape Marc	Sludge 1	Sludge 2	Sludge 3
Investment (CAPEX) – M€	3.68	3.32	3.65	3.71	3.45
Fixed costs – M€ year^{-1}	0.50	0.48	0.50	0.50	0.49
Variable costs – M€ year^{-1}	0.82	0.80	0.81	0.82	0.81
Minimum selling price – € ton^{-1}	656	1210	681	633	939
Minimum selling price – € GJ^{-1}	20.5	37.8	21.3	19.8	29.4
Gate fee – € ton^{-1} dry matter	88	117	90	85	107
Heavy Fuel Oil – € GJ^{-1}	9.4				
Vegetable Oil (Palm) – € GJ^{-1}	30				

The production costs without gate fees are in the same order of magnitude as raw vegetable oils. There remains some uncertainty on the quality of the fuel oil and its upgrading. Upgrading of raw vegetable oil is well understood. Fossil fuels are of course cheaper to produce. In all cases, this type of plant requires small scale production facilities to be close to the resources, food processing factories and population centres. Economic viability will necessarily come via gate fees to compensate for the relative small capacities, in the order of 1 to 10 ton hour^{-1}. The work shows that gate fees are comparable to current waste incineration plants, in the range of 50 to 130 € per ton of dry matter.

Conclusions

Food processing waste and sewage sludge are interesting carbonated resources. Rather than looking for low value valorisation more value can be added to these waste steams by hydrothermal liquefaction. The technology is not able to compete economically with the fossil energy industry. Most organic waste producers are used to pay to dispose of these wastes. The cost varies greatly with the nature of the waste. With gate fees in the 50 to 130 € ton^{-1} dry matter hydrothermal liquefaction can produce liquid fuels that can compete with fossil fuels. No gate fees are required to compete with raw vegetable oils. Great uncertainties subsist however about the quality of the fuels and there compatibility with existing applications.

Not all resources are however equally suited for this technology. The results are variable and optimal conditions need to be found for each resource. Lignin rich resources such as grape marc yield much lower oil yields at low temperatures. These resources should be processed at higher temperatures [23]. Resources low in organic material such as digested sewage sludge are less interesting. The oil yields are low and the biocrude is of low quality as it is very rich in inorganic material.

Sewage sludge is an interesting resource but care should be taken with the generated hydrogen sulphide. Much of the sulphur is however transferred to the water phase. The biocrude and bio-oil contain sulphur but much less that the initial resource.

Acknowledgements

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