Manure Processing Technologies
3.7 Hydrothermal Liquefaction

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What Is Hydrothermal Liquefaction?

Hydrothermal liquefaction (HTL), which is also referred to as hydropyrolysis, is a thermochemical conversion (TCC) process in which high temperatures and pressures are used to decompose complex organic material, including biomass. HTL is one of a family of thermochemical conversion processes, including pyrolysis, gasification and torrefaction, all of which use heat to chemically decompose organic material.

The major processing differences that separate HTL from other TCC processes are that it occurs at elevated pressures and that it is performed on wet biomass, using water as a critical reactant in the decomposition process.

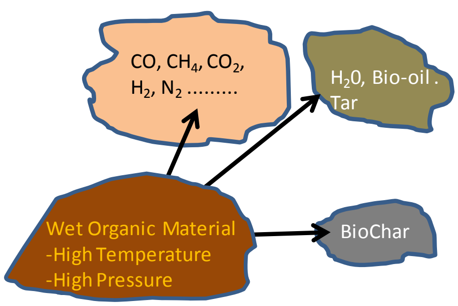

Figure 1. The HTL process.

HTL is of particular interest for development for the processing of livestock manure for a variety of reasons. First, it allows for the processing of a wet biomass, such as swine or some cattle manures, without a preliminary drying step (Ocfemia et al., 2006). Second, the conversion process transforms the manure solids into oil and gas products that can be separated from the water effluent, thereby reducing the potential environmental impact of the manure through the removal of solids (Zhang et al., 1999). Third, it produces a high energy bio oil, which has potential commercial applications both as a liquid bio fuel and as a feedstock for the development of other specialty chemical applications, such as bio-asphalt (Jena and Das, 2011)). Fourth, the high temperature and pressure destroys the pathogens present within the manure (Ocfemia et al., 2006). Currently, HTL systems remain, for the most part, under research and development, and systems for individual farm use are not commercially available. However, this technology is currently undergoing development and commercialization and may emerge into the marketplace in the future.

Feedstock Characteristics

HTL has been evaluated as a conversion technology for a wide range of biomass feedstocks, ranging from agricultural and forestry waste, sewage sludge, algae and animal wastes (Wang, 2011). These feedstocks, including animal manure, are all comprised of a complex mixture of constituent materials, including proteins and amino acids, fats, cellulose, hemicellulose and lignin. The major constituent components present in manure include protein, lipids and fibers (cellulose, hemicellulose, lignin), in addition to inorganics and/or ash. Table 1 summarizes the breakdown of these major manure components for cattle manure, swine manure, and poultry litter.

The reaction mechanisms taking place during the HTL conversion of manure are not well understood. The general pathway of conversion likely takes place via the following steps: 1) hydrolysis[1]; 2) further

[1] **Hydrolysis** is a chemical reaction in which a compound reacts with water to produce other compounds.

Table 1. Proximate analysis results for swine, cattle and chicken manure

Manure Type	Reference	Proximate analysis[1] (wt %)			
		Water Content ar	Volatile Matter d	Fixed Carbon d	Ash d
Swine	Tu et.al. 2008	68.01	70.21	12.1	17.7
Swine	ECN, Phyllis-#1366	92.1	51.3	13.3	35.4
Cattle	Tu et.al. 2008	81.4	71.24	15.38	13.38
Cattle (beef feedlot)	ECN, Phyllis-#1882	13.88[2]	70.27	13.86	15.87
Chicken	Tu et.al. 2008	73.59	61.42	10.49	28.09
Chicken (layers)	ECN, Phyllis-#3501	19.25[2]	65.56	12.80	21.65

[1]ar: as received basis, d: dry basis, [2]moisture content suggest air drying prior to collection/analysis

degradation; and 3) repolymerization[2] of some of the smaller compounds into more complex hydrocarbons (Wang, 2011). It is important to note that only the volatile, or organic, components of the manure solids can be converted to bio oil via HTL. Research has indicated that feedstocks that are higher in protein and lipid concentrations lead to a higher oil product yield, while those higher in fibrous material lead to the higher biochar yields (Wang et al., 2009; (He et al., 2001).

This correlation between protein/lipid content and bio oil yield becomes important when considering HTL processing of aged manure or manure stored in lagoons or deep pits. The properties of manure can change with age. In the example of swine (grower-finisher) manure, over a 3-week storage period, the crude protein content of the manure (with respect to dry weight) increased 87.4%, and the lipid content decreased 11.8%. In HTL studies of these fresh and aged manure samples, this change in the constituent component contents of the manure led to an approximately 27% decrease in bio oil yield (Wang et al., 2009).

One of the critical feedstock characteristics that also needs to be considered in HTL processing of manure is the moisture content. HTL processing works best with solids contents in the starting feedstock that are around 20-35 wt % (Minarick at al., 2011; He et al., 2001). Feedstocks with moisture contents significantly lower than this may prove difficult to handle, due to higher viscosities. However, manure that is too dilute will decrease the efficiency of oil production, as there will be low concentrations of volatile solids available for conversion and may require larger system sizes for conversion, increasing costs. For example, compared to a feedstock with 80% moisture, a feedstock with 90% moisture will require 2 times the volume and at 95% moisture will require over 4 times the volume to be processed.

Operating Conditions

The operating conditions that affect HTL of manure include temperature, pressure, residence time within the reactor (also referred to as retention time), pH, solids content and the oxygen to carbon ratio (Zhang et al., 1999). HTL processing typically occurs under the range of system operating conditions summarized in Table 2. A comparison between HTL processing and other thermochemical conversion technologies can be found in Table 3.

[2] **Polymerization** is a process of reacting molecules together in a chemical reaction.

Inert and/or reducing gases (i.e. N_2, CO) and catalysts have been used to improve the bio oil *yields in HTL processing of biomasses, including* livestock manure. Inert or reducing gases are used to reduce the ratio of oxygen to carbon present in the system. CO has been successfully employed for the conversion of swine and cattle manure into bio oil (Midgett and Theegala, 2007; He et al., 2001). Alkaline catalysts have also been shown to decrease char formation, particularly in feedstocks with high cellulose content (Wang, 2011). Sodium carbonate (Na_2CO_3) has been used in the HTL processing of dairy manure (Midgett and Theegala, 2007).

Table 2. HTL operating conditions (Zhang et al., 1999)

Control Factors	Typical Range
Temperature	250-400°C (482-752°F)
Pressure	5-20 MPa (725-2900 psi)
Residence time	0.2-1.5 hrs.
pH	6.5-7.5
Total solids content	20-25 wt.%
CO to volatile solids ratio	0.11

Table 3. Thermochemical conversion technologies summary

Name	Residence Time	Temp (F)	Heating Rate	Major Products
Carbonization	hours-days	572-932	very low	charcoal
Pyrolysis	hours	752-1112	low	solids, liquids, gases
Fast pyrolysis	5-30 min	1292-1652	medium	solids, gases
Flash pyrolysis	< 1 sec	1202-1832	high	liquids, gases
Gasification	10-20 sec	> 930	high	gases
Hydrothermal liquefaction	15-120 min	< 932 +high pressure	medium	liquids

Adapted from Huber et al., 2006; Klass,1998.

Laboratory-scale Processes

For the processing of swine manure, a range of conditions have been evaluated in the laboratory, with the goal of maximizing both bio oil yield and quality (defined by energy content and processability as defined by benzene solubility). Temperature ranges from 545°F to 635°F were found to produce the highest yield and quality of bio oil from fresh swine manure, with corresponding pressures ranging from approximately 1305 to 1740 psi (Ocfemia et al., 2006). Residence times of 15 to 120 minutes were found to produce bio oil, however optimum yields were identified between 15 and 60 minutes. Higher bio oil yields were found by increasing the pH of swine manure (pH of 10), however, the best oil quality was found around native pH (6.5-7) (He et al., 2001). Solids contents greater than 25% were found to be difficult to adequately pump and mix, but solids contents lower than 20% were found to have decreased bio oil conversion efficiency (He et al, 2001).

For the processing of dairy manure, HTL conditions have also been identified in the laboratory. Fresh dairy manure flushed from concrete pads was evaluated using a batch, laboratory scale HTL reactor. The manure had its moisture content adjusted to 20% for consistency, and was processed at temperatures from 482 to 662°F and at pressures from 798-2597 psi for 15 minutes, producing a maximum bio oil yield of approximately 25 wt %, with respect to initial volatile solids concentrations. In all studies, CO was used as a reducing gas to control the oxygen content in the feedstock, with 299 psi of CO initially charged in to the reactor. The use of Na_2CO_3 has also been explored for use in conversion of dairy manure and was found to increase bio oil quality. (Midgett and Theegala, 2007; Midgett, 2005; Theegala and Midgett, 2012).

For the processing of poultry litter via HTL, there has been less extensive research, due to poultry litter having other commercial and processing applications available. However, there have been some studies demonstrating that poultry litter can be readily converted to bio oil via HTL. Poultry litter can be converted using HTL processing at 662 °F and with a 15-minute residence time, using CO as a reducing gas, and using Na_2CO_3 as a catalyst. The bio oil quality and energy content were found to be slightly inferior to that produced by cattle manure processed under identical conditions, but does demonstrate the applicability of HTL processing for poultry litter as a feedstock. (Midgett, 2005; Midgett et al., 2011).

Products of HTL

HTL processing of manure yields 4 product streams: bio oil, synthesis gas (syn gas), bio char and a waste water effluent. The feedstock and processing conditions will impact the exact makeup and distribution of these product streams. It should be noted that for most HTL processes, the water content in the initial feedstock is the major component, providing at least 75% of the total mass of the feedstock, and therefore a majority of the product output is water effluent. The volatile solids content of the manure is the convertible fraction of the feedstock.(Zhang et al., 1999). An example of the representative distribution of products can be seen in Figure 2.

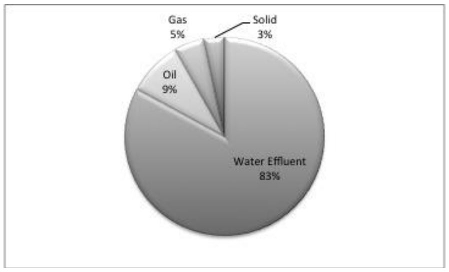

Figure 2. Product distributions from the HTL conversion of swine manure: Adapted from He, et al., 2000.

Bio Oil:

The bio oil produced from the liquefaction of swine manure is a complex mixture of hydrocarbons. The bio oils produced from liquefaction typically have a lower oxygen content and a higher heating value than those produced by pyrolysis (Huber et al., 2006). Bio oil yields have ranged from 20 wt% to 70 wt%, with respect to volatiles, depending on feedstock and processing conditions. The bio oil is mostly made up of carbon, hydrogen and oxygen, with lesser amounts of nitrogen, sulfur, ash and water. Typical properties for bio oil produced from the liquefaction of manure can be found in Table 4.

Table 4. Properties of bio oil from swine manure (Xiu et al., 2010)

Property		Bio Oil from Swine Manure
Moisture content (wt.%)		2.37
Specific gravity		1
Elemental Composition (wt.%)	C	72.58
	H	9.76
	O	13.19
	N	4.47
Ash (wt.%)		0.78
HHV (BTU/lb)		15,498.9
Viscosity (at 122°F) (cP)		843

Liquefaction bio oils have been evaluated for a variety of end uses, most commonly for use as a replacement for petroleum fuels, for either heating or transportation applications. Higher concentrations of nitrogen and sulfur in bio oils produced from HTL of manure have limited their direct use for fuel applications. However, the bio oils produced from swine and dairy manure have shown heating values upwards of 12,898 BTU/lb (Zhang et al., 1999). Additional applications have been as a replacement for petroleum derived asphalt in road construction and roofing (Fini et al., 2011). However, as with most bio oils produced via thermochemical conversion, HTL bio oils are chemically unstable without further upgrading or modification. High temperatures, oxygen and UV exposure have been found to increase the rate at which these changes can occur. (Huber et. al., 2006).

Syngas:

The syngas produced in the liquefaction of manure is mostly comprised of carbon dioxide (He et al., 2000; Zhang et al., 1999). The use of CO as a reducing process gas will also increase the amount of CO_2 produced as syn gas (Zhang et al., 1999). For most studies of HTL processing of manure, the syn gas was simply vented and not evaluated further. However, a more complete evaluation of the components within the gas stream would be required, to ensure that the production of other gases, such as methane, CO and H2S, remain at low enough concentrations to be considered negligible.

Bio Char:
The bio char, or solid residue, produced from HTL processing of manure is mostly composed of the inorganics (ash) from the starting manure, small amounts of unreacted feedstock, and char formed during the reaction. Bio char produced from HTL processing may have applications as a soil amendment, filtration media, or filler in industrial applications.

Waste Water:
The waste water effluent produced from the HTL processing of manure is mostly comprised of H_2O, which was originally in the feedstock and was generated during the process. In addition, the majority nutrients, including N, P, and K, remain with the wastewater effluents. In areas where manure nutrients can be land applied, this wastewater may serve as a nutrient source. However, in some areas this wastewater effluent may require additional treatment prior to land application or discharge.

Types of HTL Systems

To date, the majority of HTL conversion systems remain at the laboratory scale (0.26-3.96 gal.), and is used for research purposes. The bulk of these systems are stirred tank reactors, operated in batch mode. Reactor volumes for these batch systems range in scale from a few hundred milliliters (Midgett et al., 2007) to several liters (Wang et al., 2009). These reactors are typically stainless steel, with external jacket-style heaters and internal mixing. An example of a batch, laboratory scale HTL reactor can be seen in Figure 3.

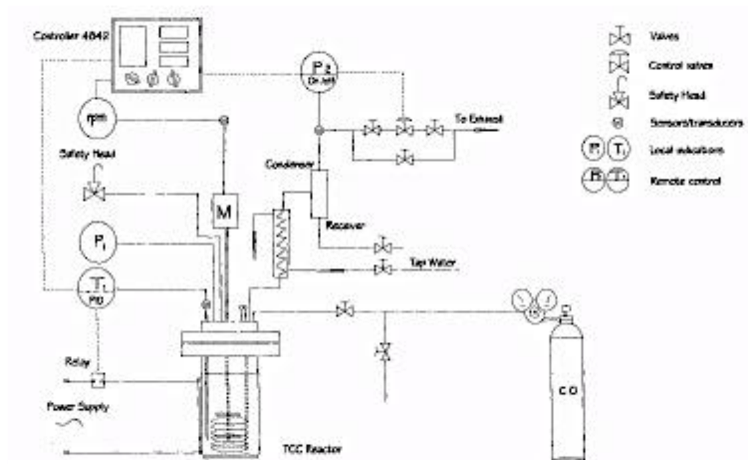

Figure 3. Schematic of a batch HTL reactor and control system. (He, et. al., 2000)

A continuous flow reactor has also been developed and evaluated by researchers at the University of Illinois (Ocfemia et al., 2006). The continuous system consisted of a 0.53 gal stirred tank reactor. The manure was fed to the reactor via a high-pressure pump and was discharged from the reactor into a separation vessel, where the gaseous products were separated from the liquid (water and oil) and char products. The reactor was capable of processing 105.8 lbs of swine manure per day, and converted manure to bio oil at yields ranging between 62 and 70.4 wt.%, with respect to initial volatile solids (Ocfemia et al., 2006). This continuous reactor can be seen in Figure 4. It is likely that for HTL to become an economically feasible process at commercial scale, continuous processing equipment like the one described here will be required.

Figure 4. Continuous hydrothermal process reactor system. (Ocfemia et al., 2006)

As of this publication date, there are no commercially available systems at any scale available for farm use, although there are companies working to develop a commercial reactor system targeting the conversion of swine manure to bio oil. Changing World Technologies (CWT), in Carthage MO, uses a similar high temperature-high pressure depolymerization process for the production of bio-diesel from poultry offal. A subsidiary of CWT, Thermo-Depolymerization Process, LLC (TDP) also developed a pilot and demonstration facility in 1999 in Philadelphia, PA. CWT had a production capacity of 400 barrels/day of bio oil. However, CWT experienced considerable challenges, both with odor control, community relations and revenue generation, and filed for bankruptcy in 2010.

HTL processing of livestock is still a developmental technology, which, for the most part, is still confined to laboratory research and small-scale systems. Challenges still exist in the development of a proven operational processing system, refining of the bio-oil, and end product uses. However, there is potential for the technology to be a valid means of manure processing for producing value-added products. Therefore, it is anticipated that as the technology matures, the agricultural community may see these processing reactors introduced in the future

What are the System Components for HTL?

The major systems required for HTL processing of manure can be broken down into the following categories: Manure Handling and Preparation; HTL System; Product Separation; Product Utilization. Due to HTL being in a relatively early stage of development, the exact requirements for these systems is undefined, however some considerations for system components are detailed in Table 5.

Table 5. Major System Component Considerations for HTL

	Process Step	Component Considerations
Manure Handling	Manure collection at farm	Storage pit, directly to digester
	Manure transport to HTL reactor	Tanker, pipeline, other
	Feedstock preparation (mixing, grinder)	Holding/mixing tank, grinder
	Feedstock dewatering	Settling tank, belt press, centrifuge, inverted screen
	Feedstock storage	Storage tank with mixing
	Feedstock Q/C equipment	pH meter, drying oven, viscometer
HTL System	HTL reactor	Feed element, heating element, high pressure/high temperature reactor, mixing for uniform heat transfer
	Control system	Required for system monitoring & operations
	Energy reclamation system	Heat capture and recycle to reduce energy requirements
	Product separation	Flash tanks, settling tanks, condensers, heat exchangers, filters
Product Utilization	Syn gas	Vent, scrub
	Bio oil	Heated storage, mixing, addition of stabilizers/anti-oxidants
	Bio char	Storage, grinding
	Waste water	Storage, land application, treatment

Daily Management of HTL systems

It is anticipated that upon commercial readiness, much of the daily management of HTL processing

systems will be automated and controlled via computer controlled programming. However, there are several considerations for daily management that should be considered, including the following:
Manure quality control: To ensure consistent conversion and product yields and quality, care should be taken to limit feedstock variability. This may come in the form of premixing manure from various ages/heights within a storage pit or lagoon, or through the introduction of other various quality control measures. However, some manual quality control testing may need to be carried out, to ensure that the manure is being introduced at the target moisture and volatile solids contents.
System maintenance: Due to the nature of the manure, care must be taken to ensure that fouling and corrosion of HTL system components are not occurring. This should be done through regular equipment inspections and cleanings. Particular care should be given to components of the feed system, due to the high viscosities that can be encountered with lower moisture manures, as well as the reactor and product separation equipment, where bio char and higher viscosity bio oil may occlude openings within the system.

What are the Benefits and Limitations of HTL?

The true scope of benefits and limitations of HTL systems for the processing of livestock manure are not fully defined at this point, as the technology is still under development. However, based on the research and pilot studies that have been carried out, one would expect to see the following:

Benefits:

- Reduces risk of water contamination from livestock manure
- Reduces risk of air contamination from livestock manure
- Reduces the need to pre-dry the manure before processing/applicable to wet feedstocks
- Produces products (bio oil, bio char) with potential market value
- Has the potential to create a product (bio oil) which can serve as a replacement for petroleum products (pending future research & development)

Limitations:

- Technology is still under development
- Markets for products are still under development
- Requires high pressures – leading to possible safety concerns and higher equipment capital costs to ensure pressure tolerance
- Requires relatively high temperatures – leading to possible safety concerns and relatively high energy costs
- Does not fully remove all manure nutrients (NPK) from the waste-water effluent – further treatment may be required for disposal
- Product separation may prove challenging at scale
- Some dewatering may be required if processing low solids content manure (i.e. manure stored in deep pits) – leading to added capital and operation costs

What is the Income Potential of HTL?

As HTL systems are not yet commercial, it is difficult to quantify the economics of a farm-scale system. However, researchers in this field have conducted some estimates of the economics of implementation and operation of HTL conversion systems. Minarick et al. (2011) has also conducted theoretical estimates on the economics of a farm scale HTL system for the conversion of deep pit manure. In particular, this work compared the use of different liquid-solid separation equipment as a means of dewatering deep pit manure. This work served to estimate the net present value of HTL conversion by considering the energy balance, energy costs associated to achieve conversion (assuming 75% efficient heat recovery system), bio oil yield and quality, bio oil potential market value, waste water effluent market potential (as liquid fertilizer), and annual labor costs (estimated at $35,000 per 10,000 hogs per year). Not considered in Minarick et al.'s study, but what must be accounted for are equipment capital and maintenance costs; product storage, transportation and other logistical costs; and waste disposal/treatment and permitting costs. Several key technical and economic factors that should be considered in evaluating the costs and income potential of HTL processing of manure can be found listed in Table 6.

Table 6. Technical and Economic Considerations for HTL Processing of Manure (Minnarick et al., 2011)

Capital Costs	Manure handling/transportation equipment
	Manure preprocessing/drying equipment
	HTL reactor
	Heat recovery system
	Product separation equipment
	Product storage
	Product transportation equipment
Operating Costs	Energy (Affected by water content, operating conditions and energy recovery)
	On site labor
	Transportation
	Waste treatment/disposal
	Permitting
	Maintenance
Revenue Streams	Bio oil
	Bio char
	Waste water effluent
Other Technical Considerations	Bio oil energy value
	Energy value of feedstocks
	Heat/energy losses in HTL system
	Heat recovery system efficiency
	System operating conditions to maximize product value & minimize operating costs
	Energy cost per kWh (use of natural gas vs. electric vs. other)
Other Economic Considerations	HTL product market price
	Equipment life expectancy

Additional Resources

Hydrothermal Liquefaction Process Strategy. National Advanced Biofuels Consortium
http://www.nabcprojects.org/hydrothermal_liquefaction.html

Hydrothermal Liquefaction: A Route to Improved Bio Oils. National Advancement Biofuels Consortium.
http://www.nabcprojects.org/pdfs/hydrothermal_liquefaction_route_to_improved_bio-oils.pdf

Hydrothermal Liquefaction to Convert Biomass into Crude Oil, Yuanhui Zhang. Book chapter in Biofuels from Agricultural Wastes and Byproducts. http://age-web.age.uiuc.edu/bee/research/IntroHTL.pdf

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