

## Bio-Energy Generation from Food Waste by Hydrothermal Liquefaction

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### Abstract:

Growing nation has a great need for energy. Energy demand is severed by many environmental concerns due to increasing carbon emissions led by burning of fossil fuels. There are numerous ways being followed to generate energy from food waste. This paper attempts to introduce various technologies for conversion of waste to energy and a detailed analysis on the technique of Hydrothermal Liquefaction of food waste considering a model food waste sample. Thus, finally the end products of the process are further discussed to obtain electrical energy. Hydrothermal Liquefaction mimics the natural process of fossil fuel formation from biomass feedstocks and it holds great promise as a renewable energy source.

**KEYWORDS:** Hydrothermal Liquefaction, fossil fuel, biomass, renewable energy.

### Introduction:

As population and urbanization grows, more food is being produced and more food is being wasted. Moreover, food wasted in an urban context creates severe environmental and public health consequences that have a negative impact upon human well-being and the environment. Hyderabad residents generate the highest per capita waste in India - 570 gm per day [1].

Energy can be generated from such wastes and is known as Waste-to-energy. The energy can either be electric or heat energy. It is possible to valorize a greater fraction of energy from food waste through numerous processes [2]. Hydrothermal liquefaction is a thermal depolymerization process used to convert wet biomass into crude-like oil -sometimes referred to as bio-oil or biocrude- under moderate temperature and high pressure [3]. A number of studies on individual components of food waste have been performed (e.g., glucose, amino acids, fatty acids), but there is minimal data available using food as a feedstock to produce bio-oil. While most thermochemical conversion processes require a relatively dry feedstock, hydrothermal liquefaction uses subcritical water as the reaction medium and is therefore compatible with high-moisture feedstocks [4].

### Materials and Methods:

A wide range of treatment technologies for solid biowaste already exist and have been extensively researched over the last decades [5]. All these treatment technologies can convert organic waste into a variety of output products with more or less market value and ecological benefits. The categories of technologies are:

- (1) direct use.
- (2) biological treatment.
- (3) physio-chemical treatment.
- (4) thermochemical treatment.

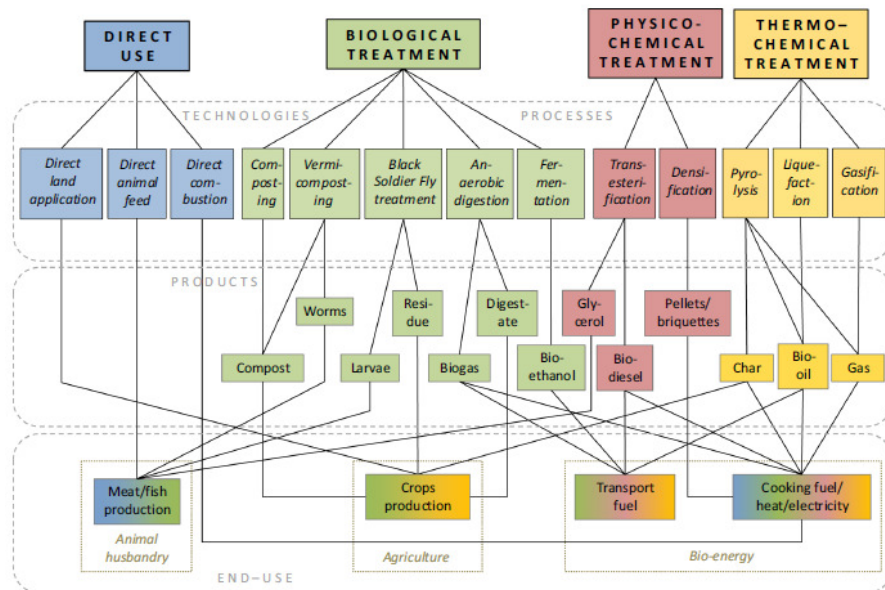


Fig. 1. Overview of biowaste treatment technologies as presented in this review with the respective products generated from waste and their end-use [6].

### Waste to energy (wte) Techniques:

There are number of processing techniques for conversion of waste to energy (WtE). As an example of energy recovery, anaerobic digestion has been practiced for decades to generate methane for onsite heat and/or electricity generation, and some facilities have achieved or neared a net-zero energy footprint.

#### Thermal Technologies:

##### A. Incineration

Incineration generally entails burning waste (residual MSW, commercial, industrial and RDF) to boil water which powers steam generators that generate electric energy and heat to be used in homes, businesses, institutions and industries.

##### B. Gasification

Gasification produces combustible gas, hydrogen, synthetic fuels.

##### C. Pyrolysis

Pyrolysis produces combustible tar/bio-oil and chars.

##### D. Thermal depolymerization:

Thermal depolymerization produces synthetic crude oil, which can be further refined.

##### E. Plasma Gasification process

Plasma gasification process (PGP) produces rich syngas including hydrogen and carbon monoxide usable for fuel cells or generating electricity to drive the plasma arch, usable vitrified silicate and metal ingots, salt and Sulphur

#### Non-thermal Technologies:

A. Anaerobic digestion: Biogas rich in methane

B. Fermentation production: examples are ethanol, lactic acid, hydrogen

C. Mechanical biological treatment (MBT)

MBT + Anaerobic digestion

MBT to Refuse derived fuel

Among all these the Hydrothermal Liquefaction(HTL) plays a crucial role in converting the biomass to energy. This process diverts organic wastes, including wastewater sludge, livestock waste, and food waste, for beneficial energy use while reducing the quantities of waste that are disposed or released to the environment [7].

*Hydrothermal Liquefaction:*

HTL mimics the natural process of fossil fuel formation from biomass feedstocks. In that sense, it holds great promise as a renewable energy source, especially for liquid fuel. Although great effort has been made since the 1970s, the results have been intermittent due to the low energy price since the 1980s. It is time once again that we need to revisit the science and technology of HTL, together with other renewable technologies [8].

The HTL process has been shown to produce bio-oils with energy densities between 3537 char with an energy density around 28 MJ/kg which is similar to that of coal (Vardon, 2012). The hydrothermal liquefaction of biomass has been previously shown to produce more energy than it consumes (Gollakota, 2017). This means that the process could be run by burning part of the oil/ char it produces and have a percentage left over. The percentage remaining can even be chemically upgraded to produce transportation fuels.

HTL of biowaste materials into liquid fuel holds several unique advantages. First, it safeguards national security by using biowaste and producing algae within dispersed local communities, the geopolitical threat from the required importation of petroleum can be neutralized. Second, it has a net - zero carbon emission bio - waste materials are renewable, typically a product or byproduct of photosynthesis. Third, it does not compete with the food supply bio - waste is often a negative - cost feedstock [9].

The principle behind it is, the bio oil is produced is the reduction of oxygen composition from the biomass feed. Oxygen accounts for 40-60% of the dry weight of biomass (Peterson, 2008). This is done by reducing the number of oxygen molecules bound to the organics and increasing the organic molecules size. The reduction of bound oxygen reduced the solubility of the organic compound by making it less polar and more hydrophobic. This reduction in oxygen increases the energy density of the resulting bio oil. Two major chemical reactions taking place are

Alkylation of phenolic compounds and

Ketonization of carboxylic acids.

Removing oxygen in these ways is ideal for the process as it either removes it as water, increasing the total carbon yield, or as carbon dioxide which increases the hydrogen carbon ratio of the final products (Peterson, 2008). Bio-oil products of the HTL process on average are less than 1% oxygen. The addition of catalyst has been shown to improve the percent conversion from the aqueous to oil phase [10].

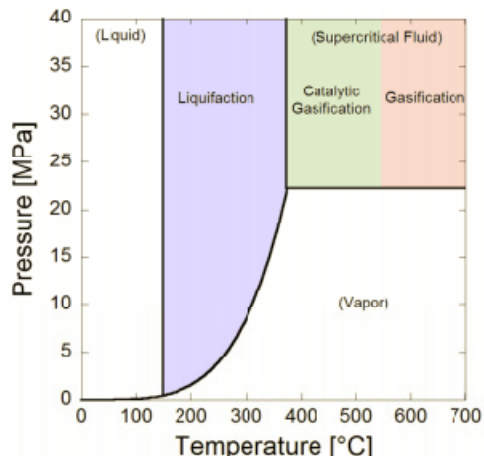


Fig. 2. Effective reaction temperature zones [9].

### Previous Works:

One of the first HTL studies was conducted by Kranich (1984) using MSW as a source to produce oil. Three different types of materials from MSW plant were used: primary sewage sludge, settled digester sludge, and digester effluent. Temperatures ranged from 295–450°C with pressures up to 14MPa. Retention times also varied between 20–90 minutes. Hydrogen was used as the reducing gas with initial pressures up to 8.3MPa. Three types of catalyst were studied: sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), nickel carbonate ( $\text{NiCO}_3$ ), and sodium molybdate ( $\text{Na}_2\text{MnO}_4$ ). Gas products were found to contain  $\text{H}_2$ ,  $\text{CO}_2$ , and C1–C4 hydrocarbons. The experimental results showed no significant differences between the applications of the three different catalysts. Kranich's recommendation did not hold, mainly due to increases in crude oil prices and the need to find new technologies for energy procurement, and thus many studies on liquefaction of sewage sludge have since been conducted. Research has indicated that liquefaction is a feasible method for the treatment of sewage sludge wastes and has a high oil producing potential (Molten, 1985; Suzuki, 1986; Itoh, 1994; Inoue, 1997; and Furness, 2000). Today HTL research is still being conducted with sewage sludge; however, focus has shifted to include many varieties of biomass materials.

Ogi et al (1985) studied the effect of various salts and bases ( $\text{CaCO}_3$ ,  $\text{Ca}(\text{OH})_2$ ,  $\text{NaOH}$ ,  $\text{HCOONa}$ ,  $\text{NaCl}$ ,  $\text{K}_2\text{CO}_3$ ,  $\text{KOH}$ , and  $\text{HCOOK}$ ) at temperatures of 300–350°C and discovered that up to 50% heavy oil-like products were obtained in the presence of base catalysts. Nickel catalyst, was used by Fang et al (2004), nickel promoted conversion to gas (74%) instead of oil; while  $\text{Na}_2\text{CO}_3$  yielded an oil fraction of 43% on an organic basis.  $\text{RbOH}$  and  $\text{CsOH}$  were used for the catalytic hydrothermal treatment of pine wood (Karagoz, 2005). Again, both base catalysts hindered the formation of char and favored the formation of oil products [5].

The hydrothermal liquefaction of food waste has been studied using many different catalysts. One particular study compared the effectiveness of a Sodium Carbonate ( $\text{Na}_2\text{CO}_3$ ) catalyst to a Ceria Zirconia ( $\text{CeZrO}_x$ ) catalyst to convert food waste to bio oil. Many catalysts used for this process are alumina, iron oxide, calcium oxide, red mud along with the pure thermal method [11].

The average higher heating value of the HTL oil, generated at the ideal conditions for the surrogate food waste, was 35.1 MJ/kg. Process conditions using a blanket of air at atmospheric pressure yielded similar quality oil products compared to using syngas. Solids loading and residence time also did not appear to have a significant effect on the energy content of the oil. Comparisons between the raw starch and casein feeds and their respective bio-oil clearly show upgrading occurred. Elemental analysis demonstrates that the oxygen content of these feedstocks is reduced by over 50% after reaction. Also, the concentration of carbon increased drastically for both feeds. Reaction of soybean oil, based on both the heating value and elemental analysis, resulted in no upgrading of the raw feed. No appreciable reaction appeared to occur as the elemental composition remained unchanged [12].

Hydrothermal liquefaction (subcritical state: 280e380 °C and 7e20 MPa for 10e60 min) produces a biocrude oil with lower oxygen content (10e20 wt%) and higher energy value (30e35 MJ/kg). The

relatively low oxygen content leads to improved thermal stability and lower associated upgrading costs. Furthermore, HTL is carried out in presence of water, thereby making use of biomass moisture to avoid the high energy- and capital-intensive drying step required in pyrolysis. This HTL process can also be performed by a range of different catalysts, varying from alkaline solutions to solid iron-based materials [13]. Hydrothermal liquefaction (also known as direct liquefaction) is essentially pyrolysis in hot liquid water. As such, it does not require a catalyst, but a significant amount of research and development on catalytic methods in hydrothermal liquefaction has been undertaken. The most commonly considered “catalyst” has been the use of alkali to modify the ionic medium to favor certain base-catalyzed condensation reactions, which can lead to aromatic oil formation, in preference to acid-catalyzed polymerization reactions, which lead to solid product formation. While HTL proceeds through pyrolytic mechanisms, its biocrude product is much different from fast pyrolysis bio-oil. It is more deoxygenated through, among other reaction pathways, condensation reactions of the light fragments generated from the biomass, resulting in a more hydrophobic phase with less dissolved water. Physically, the biocrude is much more viscous, but is actually less dense than bio-oil. Analysis of the HTL products from woody biomass show the biocrude product is clearly not a petroleum analog. It is a complex mixture of oxygenated compounds extending over wide range of molecular weight [14].

The use of a nickel catalyst could catalyze the steam reforming reaction of aqueous products as intermediates and the methanation reaction. However, nickel or metal-based catalysts tend to promote gas production instead of oil production. Proposed reaction model of the role catalysts may play in the conversion of cellulose to oil. It is important to note that hydrolysis plays an important role in the decomposition of cellulose to sugar; Minowa states that hydrolysis is the first reaction that occurs during the liquefaction of cellulose as well.

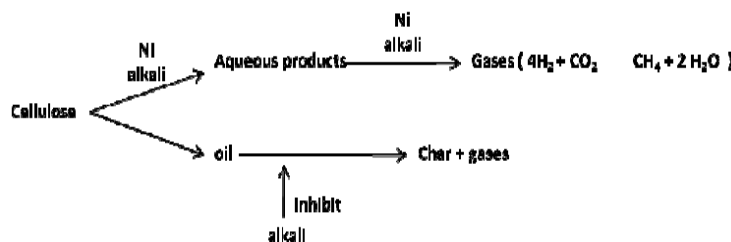


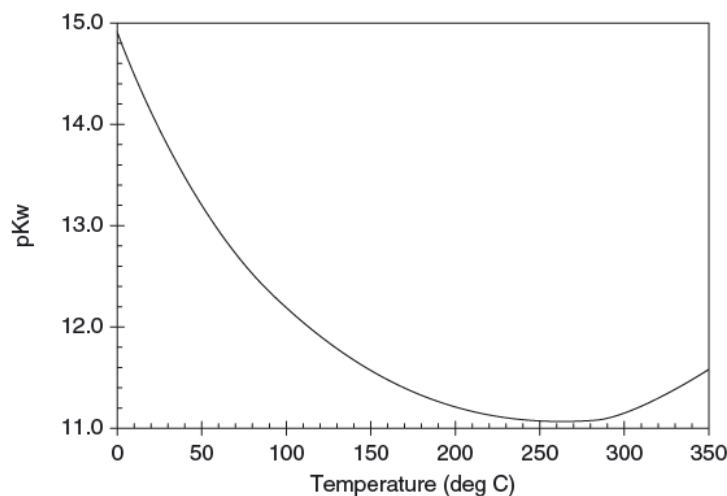
Fig. 3. Proposed reaction of metal and base catalysts roles during an HTL operation. (Minowa,1997) [5].

### The Role of Water in HTL:

Water plays an essential role in HTL. It is therefore critical to understand the fundamentals of water chemistry when subjected to high temperature conditions. Water is rather benign and will not likely react with organic molecules under standard environmental conditions (20°C and 101,325 kPa). However, when the temperature increases, two properties of water molecules change substantially. First, the relative permittivity (dielectric constant), of water decreases quickly when the temperature increases. When the thermal energy increases, the shared electron by oxygen and hydrogen atoms tends to circulate more evenly and the electronegativity of the oxygen molecule is reduced (less polar). For example, when temperature increases from 25 ° C to 300 ° C, the relative permittivity decreases from 78.85 to 19.66, resulting in water molecules from very polar to fairly nonpolar, in a relative term. This polarity Hydrothermal Liquefaction to Convert Biomass into Crude Oil change makes water more affinitive to the organic hydrocarbons, most of which are nonpolar molecules. Second, the dissociation of water dramatically increases with the increase of temperature. Water, like any other aqueous solutions, split into H + and OH – ions in hydrolysis or dissociation. This process is reversible and the rate is sufficiently rapid so it can be considered to be in equilibrium at any instant. Based on Arrhenius reaction rate, the equilibrium constant (or the dissociation constant),  $K_w$ , affected by the temperature change, can be written as (Benjamin 2002):

$$\frac{K_{w1}}{K_{w2}} = \exp \left[ \frac{\Delta E_{Ar}}{R} \left( \frac{1}{T_1} - \frac{1}{T_2} \right) \right]$$

where  $K_{w1}$  and  $K_{w2}$  are equilibrium constant at temperatures  $T_1$  and  $T_2$ , respectively;  $\Delta E_{Ar}$  is the net change in heat content of the molecules in the overall reaction, also called the molar enthalpy of reaction;  $R$  is the universal gas constant, and  $T$  is the absolute temperature in Kelvin.  $\Delta E_{Ar}$  is an empirical constant specific to particular reaction and in units of energy per mole. The effect of temperature on water dissociation is illustrated in Fig.4, where the left side vertical axis is in  $pK_w = -\log_{10}(K_w)$ , and the right side vertical axis is the ratio of  $K_w$  to the  $K_{w0}$ .  $K_{w0}$  is the water dissociation constant at a temperature of 25°C at atmospheric pressure, and is  $10^{-14}$ . From Fig.4, water molecules dissociation constant at 300°C is about 500 times higher than that of 25°C at atmospheric pressure. The increase in the dissociation constant will increase the rate of both acid and base-catalyzed reactions in water far beyond the natural acceleration due to increased temperature [15].



**Fig. 4.** Effect of temperature on water dissociation constant at 25 MPa. The dissociation constant  $K_w$  is expressed as  $pK_w$ , where  $pK_w = -\log_{10}(K_w)$ .

There is also a (usually small) dependence on pressure (ionization increases with increasing pressure). The dependence of the water ionization on temperature and pressure has been fully investigated (Fig 3) and a standard formulation exists (IAPWS 2004). For the above two reasons, water at high temperatures becomes a good solvent for hydrocarbons that are typically nonpolar hydrophobic under standard environmental conditions. These changes in physical properties make the solvent properties of water at 300 °C roughly equivalent to those of acetone at 25 °C. Ionic reactions of organics should be favored by increased solubility in water. The enhancement of this solubility of hydrocarbons in water will further enhance the possibilities of contact of dissociated  $H^+$  with hydrocarbons, hence accelerate the activities of hydrolysis. The acidic and basic nature of hot water — rather than heat — drives this cascade. For example, water may function first as a base, nibbling away at certain linkages in the organic material. As new molecular fragments build up and modify the reaction environment, water can change its catalytic nature. It can then act as an acid, accelerating different reactions. The resulting products attack parts of the remaining molecules, further speeding the breakdown (Siskin and Katritzky 1991). The above analysis may also help to explain why HTL will more likely directly convert biomass into oil than pyrolysis in which water is not involved [15].

HTL can convert lignocellulose into oil, but the yield is relatively low when no catalyst or solvent is used. Wang et al. (2008) compared the oil produced from different biomass including legume straw, corn stalk, cotton stalk, and wheat straw — under hydrothermal conditions of temperature 350 °C, residence time 2 – 3 hours, solid content 15% (dry mass), and pressure 10 – 13 MPa without using any catalyst. Hydrothermal experiments were carried out with 5.0 g biomass in a stainless tubular reactor which is 100 mm length by 10 mm internal diameter. The heating rate of reactor is about

10°C/min. The oil product was separated from reaction mixture by distillation at 101 – 405 ° C and atmospheric pressure. Water and oil automatically separated into two phases in their study. Experimental results showed that oil yield is in the range of 5.2% – 10.5% and both char and gas yield are more than 35% as the total biomass was almost completely converted. In addition to CO<sub>2</sub>, gas products contained about 4.4% – 8% H<sub>2</sub> and 5.5% – 13.3% CO. Analysis of the oil product indicated that oil mainly consists of alkanes, cycloalkanes, and aromatic hydrocarbons. Based on these results, they concluded that the component of starting material had little effect on oil composition [16].

Hydrothermal liquefaction (HTL) is a thermal process that converts biomass to several products including an oil portion with high heating value (Alba et al. 2012; Biller and Ross 2011; Brown et al. 2010; Duan and Savage 2011; Jena and Das 2011; Jena et al. 2011; Minowa et al. 1995; Vardon et al. 2011; Yu et al. 2011). HTL offers several advantages compared to other approaches. HTL is a liquid-phase process that avoids the energy cost of vaporizing the process slurry [17].

The majority of HTL research has focused on documenting the process conditions that maximize bio-oil yields. Little to no attention has been placed on HTL co-products such as the aqueous phase and the hydro-char [8]. Hydro-char is often overlooked as a by-product. However, its chemical characterization is important for determining HTL reaction mechanisms and chemical pathways at different temperatures and reaction times. The goal of this study is to expand on existing work performed in HTL, using representative waste feedstocks from a variety of industries: dairy industry (manure and yogurt whey) municipal wastewater treatment, food industry (dining halls, apple cider, wine, whiskey and olive oil). Specifically, this work emphasized characterization and quantification of mass yields, along with chemical compositions of the hydro-char by-products using FTIR coupled with PCA. To the best of our knowledge, this is the first study entirely focused on hydro-char produced by HTL processing of a diverse set of waste streams. Improved characterization of these hydro-char could potentially expand their range of applications and opportunities beyond soil remediation or the production of a solid fuel [18].



Fig. 5. Conversion of Wet Biomass to Hydro-char.

**Procedure:**

The process of hydrothermal liquefaction was carried out for a model food waste sample, which comprises of sample components of some foods. Then the yield was analyzed to obtain the bio-fuel, which was in different phases like gas, char and oil.

**Food waste slurry preparation:**

The food waste slurry was modeled after the slurry used by Mainstream Engineering. The feedstock was made up of common food items that include proteins, carbohydrates, fruits, vegetables, dairy, and fat. The food was mixed with DI water using a blender. The mixture was then stored in a jar between runs. For each run 100 grams of the mixture was combined with 5 grams of catalyst. The exact mixture is listed in Table 1 below.

Table 1. Model food waste sample formulated with various items.

S.no	Food waste	Amount (%)
1.	Cheese	12.8
2.	Chicken breast	14.9
3.	Brown gravy	2.1
4.	Mashed potatoes	10.6

5.	Green beans	14.9
6.	White rice	19.1
7.	Apple dessert	22.3
8.	Butter	3.2

### Reactor conditions:

The hydrothermal liquefaction experiments were carried out in a 300 mL Parr stainless steel reactor rated at 3500 psi and 350 °C. The reactor was sealed with a Teflon O-ring and two split ring closure clamps. The head of the reactor is equipped with a magnetic motor, pressure gage, thermocouple, pressurization valve, and emergency rupture disk. The temperature and the magnetic stirring motor were controlled by a Watlow temperature controller. The reactor was heated using an external heater. Simultaneously the magnetic stirrer was cooled by a VWO Scientific Products Water Circulator to ensure it did not overheat. The pressure of the reactor was monitored using a pressure gauge installed on the reactor head [9]. The set up can be seen in Fig. 6.

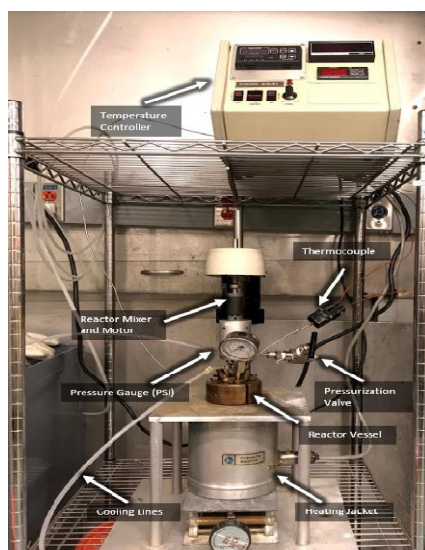


Fig. 6. Reactor Setup.

### Retrieval and Analysis:



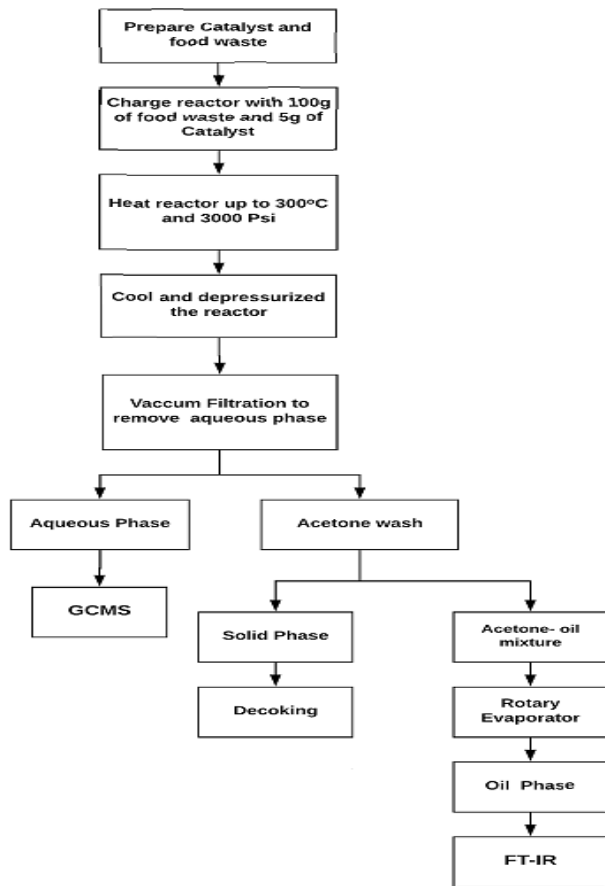


Fig. 7. Experimental and Analytical Procedure.

**YIELDS:**

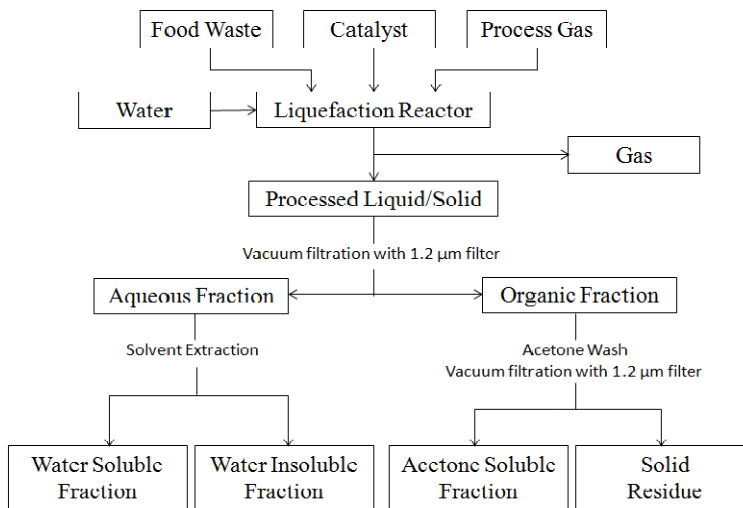


Fig. 8. Flow-Chart showing the procedure of Hydrothermal Liquefaction and the types of yields obtain.

**Results:**

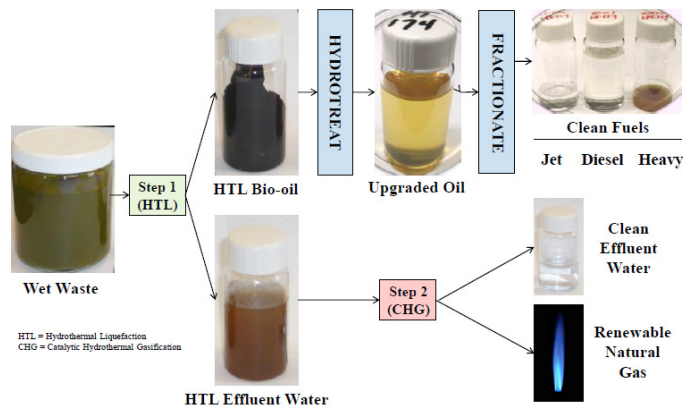


Fig. 9. Results of Hydrothermal Liquefaction and Gasification of wet waste.

**Chemical composition:**

Diesel is mainly composed of alkanes, alkenes and aromatics, while biodiesel is more oxygenated, comprised of fatty acid methyl/ethyl esters. HTL bio-crude, on the other hand, is a complex mixture of oxygenated organic chemicals, aliphatics, sugars, oligomers, nitrogenous aliphatics, and nitrogenous aromatics [19]. Table 2 shows the main chemical groups for bio-crude.

Table 2. Major Chemical Groups of Bio crude [20].

	Main Components	Area% * Range
1.	Phenolics	6%–65%
2.	Esters	2%–44%
3.	Aromatics and Heterocyclics	6%–35%
4.	Aldehydes	0%–18%
5.	Carboxylic acids	2%–40%
6.	Ketones	0%–38%
7.	Alkanes	9%–13%
8.	Nitrogenates	12%–23%

NOTE: Area % from gas chromatography-mass spectrometry results.

**Upgrading processes:**

Due to the similarity of pyrolysis and HTL, recent research on HTL bio-crude upgrading has so far focused on upgrading technologies that had previously been studied for pyrolysis bio-oils. The upgrading processes for pyrolysis bio-oils were themselves inspired by petroleum refining technologies. Although the authors draw on learnings from the pyrolysis literature, we have applied the understanding to HTL conversion. Upgrading processes discussed in here are shown in Fig. 10.

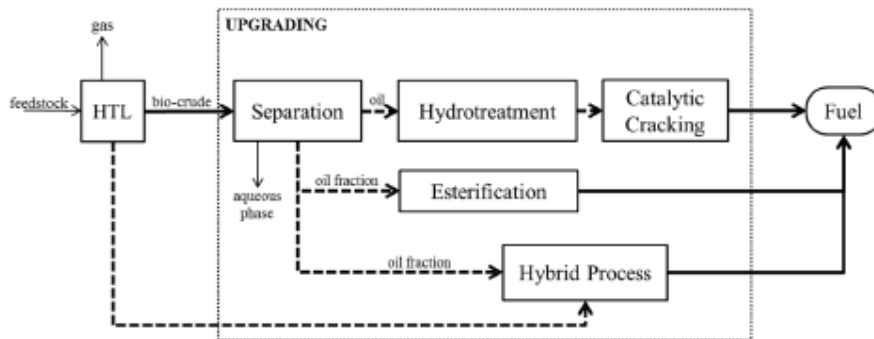


Fig. 10. Upgrading process of HTL.

**Key fuel properties:**

These final fuel properties may not be directly influenced by upgrading processes; however, some consideration should also be given to improving them when processing bio-crude. Brief discussions of some key fuel properties to be considered are provided here.

**Table 3.** Properties of various chemical groups and their effect on diesel properties [21].

Group	Ignition Quality	Heating Value	Density
n-Alkanes	Good	Low	Low
Isoalkanes	Low	Low	Low
Alkenes	Low	Low	Low
Cycloalkanes	Moderate	Moderate	Moderate
Aromatics	Poor	High	High

**Outcomes:**

Yields were calculated as follows:

$$\text{Gas yield} = (\{\text{mass of gas collected}\} - \{\text{mass of process gas feed}\}) / \{\text{mass of dry biomass}\}$$

$$\text{Char yield} = \{\text{mass of solid residue}\} / \{\text{mass of dry biomass}\}$$

$$\text{Oil yield} = (\{\text{mass of ASF}\} + \{\text{mass of WISF}\}) / \{\text{mass of dry biomass}\} [22].$$

**Benefits:**

The HTL process has the following benefits:

Crude HTL oil has high heating values of approximately 35-39 MJ/kg on a dry ash free basis

The HTL process only consumes approximately 10-15% of the energy in the feedstock biomass, yielding an energy efficiency of 85-90%

Crude HTL oil has very low oxygen, Sulphur and water content (compared to e.g. pyrolysis oil which typically contains approx. 50% water)

HTL oil recovers more than 70% of the feedstock carbon content (single pass)

HTL oil is storage stable, and has comparatively low upgrading requirements, due in part to a high fraction of middle distillates in the crude oil [23]. It is much less upgrading intensive than e.g. pyrolysis oil, which needs immediate upgrading in order not to deteriorate [24].

**USES:**

The bio-oil from HTL can be used as-produced in heavy engines or it can be hydrogenated or thermally upgraded to obtain diesel-, gasoline- or jet-fuels by existing refinery technology. In this sense, HTL bio-oil is directly comparable to fossil crude oil. This is unique among liquid bio-fuels and means that it can directly enter the existing fuel distribution network for automotive transportation in any concentration, giving it full drop-in properties [25].

**Electricity out of the yield:**

Three technologies are used to convert oil into electricity:

A. Conventional steam – Oil is burned to heat water to create steam to generate electricity [26].

B. Combustion turbine – Oil is burned under pressure to produce hot exhaust gases which spin a turbine to generate electricity [27].

C. Combined-cycle technology – Oil is first combusted in a combustion turbine, using the heated exhaust gases to generate electricity. After these exhaust gases are recovered, they heat water in a boiler, creating steam to drive a second turbine [28].

## **Conclusion:**

The current review on continuous HTL of biomass leads to the conclusion that there is significant potential for commercialization of the technology. Several feedstocks have successfully been processed at high feed concentrations resulting in high energy recoveries and carbon efficiencies. HTL mimics the natural process of fossil fuel formation from biomass feedstocks. In that sense, it holds great promise as a renewable energy source, especially for liquid fuel.

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