

Hydrofaction™ Renewable Crude Oil Upgrading Pathways to Finished Fuels and Refinery Integration

Julie Katerine Rodriguez Guerrero¹, Sergios Karatzos¹, Bob Moll¹, Andrew Ironside¹, Steen Iversen²

¹Steeper Energy Canada Ltd, Suite 220, 906 12th Avenue S.W. Calgary T2R 1K7, Canada. E-mail: jkr@steeperenergy.com

²Steeper Energy Canada Aps, Sandbjergvej 11DK 2970 Hørsholm Denmark.

Abstract

Renewable crude oil produced from woody biomass via Hydrofaction™ has low oxygen and high calorific value compared to other bio-oils, and in many ways resembles its fossil counterparts. However, it has distinct properties that need to be addressed during the upgrading process. Such properties include high viscosity and Total Acid Number, oxygen content around 10 wt.% and low sulphur content. An extensive program for characterizing and upgrading the biocrude to “Drop-in” transportation fuels or blend stocks has been performed. Different upgrading strategies including stand-alone and co-processing to maximize the yield of renewable diesel have been employed.

1. Introduction

Efficient and economic utilization of biomass and organic waste resources for the production of liquid biofuels has become attractive for industry and governments that wish to decarbonize long-haul transportation. Renewable crude oil produced from woody biomass via Hydrofaction™, Steeper Energy’s proprietary hydrothermal liquefaction (HTL) technology, needs to be upgraded/refined before it can be used in transport fuel applications. Properties that need to be addressed during the upgrading process include: high viscosity, high Total Acid Number (TAN), oxygen content (around 10 wt.%) and low sulphur content. These properties can lead to operational challenges such as reactor temperature control; upgraded oil recycling; considerable decrease of viscosity after oxygen and TAN reduction and, deactivation of commercial sulphided catalysts due to low sulphur content of the biocrude (<200 ppm). An extensive program for characterizing and upgrading the biocrude oil to “Drop in” transportation fuels or blend stocks has been performed using facilities at University of Calgary, University of Alberta as well as Aalborg University. One of the aims of this upgrading program has been to employ different upgrading strategies including stand-alone and refinery co-processing in order to maximize the yield of renewable diesel that is on or close to specification. The presentation will cover the main results to date from these upgrading strategies including longer-term stability tests (>700 hours). These results demonstrate the upgradability of the oil with both commercial and novel (biocrude-adapted) catalysts. Full upgrading with commercial catalysts, for example, achieved a doubling of the distillation fraction BP <350 °C, and the residue (BP > 550 °C) was reduced from 28% to 7.9% mass. TAN and oxygen content are eliminated, and the hydrogen to carbon ratio has been increased from 1.25 to 1.73. The diesel and heavy (350+°C) fractions from upgrading were used to produce various diesel and marine fuel blends based on predictions from optimized blending models for compliance with the EN590 (2013) ultra-low sulphur diesel standard and ISO 8271 (2012) specifications for low sulphur marine fuel oil.

2. Fundamentals of Hydrofaction™

Steeper Energy’s proprietary hydrothermal liquefaction technology, Hydrofaction™, converts biomass wastes to liquid transportation fuels in a cost-effective way. HTL is a thermochemical method for direct conversion of organic materials to renewable crude oil in high pressure water

near its critical point as the reaction medium for the conversion. Under these conditions, water obtains special properties that make it an ideal medium for chemical reactions such as acid and base catalyzed reactions as well as radical reactions. Furthermore, the properties are tunable by controlling operational conditions such as pressure and temperature.

Hydrothermal Liquefaction is considered a resource-efficient technology due to very high conversion and high carbon efficiency into the desired product. By contrast, biological processes such as Lignocellulosic ethanol and bio-gasification or anaerobic digestion do not convert the lignin which represents about 30-45% of the carbon and 35-55% of the energy contained in the feedstock [1],[2]. Unlike other thermochemical methods such as gasification/Fischer-Tropsch and pyrolysis, HTL requires no drying of the feedstock due to absence of phase change. Thus, no energy is consumed due to latent heat addition which allows processing of wet materials. Furthermore, HTL has a high energy efficiency as it is a direct conversion process with high carbon efficiency, few processing steps, and extensive heat recovery. In fact, HTL has been recognized by US DOE as being exceptionally cost- resource- and energy-efficient with the highest GHG mitigation, especially in heavy and long-haul transport [3]. Hydrofaction™ technology applies a few features that differ from other HTL process and allow for having a higher yield, higher crude oil quality, and improved energy efficiency:

- Operational conditions above the critical point of water at relatively high temperatures (390-420 °C) and pressures (300-350 bar);
- Use of homogeneous catalyst in the form of potassium carbonate and capping agents;
- Recovery and recirculation of catalysts, water soluble organics and oil for improved feed characteristics, energy balance and yields as well as improved process economics;
- Alkaline conditions for desired catalytic effects and minimizing of corrosion;
- High dry matter content slurries.

Steeper Energy aims for production of renewable “Drop In” liquid transportation fuels for long-haul transport (i.e. trucks, aviation and marine ships), where electrification is not a viable option. Further, despite that Hydrofaction™ has inherent feedstock flexibility [2],[3], the initial focus for commercialization is on woody biomass (forestry- & sawmill) residues including bark & branches as it is by far the most abundant biomass resource [6]–[10] and has formed the basis for most of Steeper’s efforts to date. Wood species tested include spruce, pine, birch, energy wood (road cuttings), coppiced willow and combinations thereof. Key results are described in detail in the references [11]–[17]. The Hydrofaction™ process block diagram platform (Figure 1) includes two stages: HTL process followed by hydroprocessing of the biocrude.

The mass and energy balances reported in Table 1 are based on data collected during steady-state operation of the Hydrofaction™ plant (Figure 2) at operational conditions described above and using a 50/50 wt.% wood mixture of spruce and pine, listed in [15]. The average values are given in the table below together with the elemental balances for carbon, oxygen, and hydrogen. It is worth noting that the overall mass balance is determined to be on average 100.3 wt.% indicating that no mass is accumulated within the plant. The results show that the Hydrofaction™ process has a mass yield of about 45 wt.%, a carbon recovery in the oil of more than 73 wt.% and an average energy recovery of 85%.

Figure 1: Process block diagram of the Hydrofaction™ platform consisting of HTL stage and subsequent hydroprocessing stage. [13]

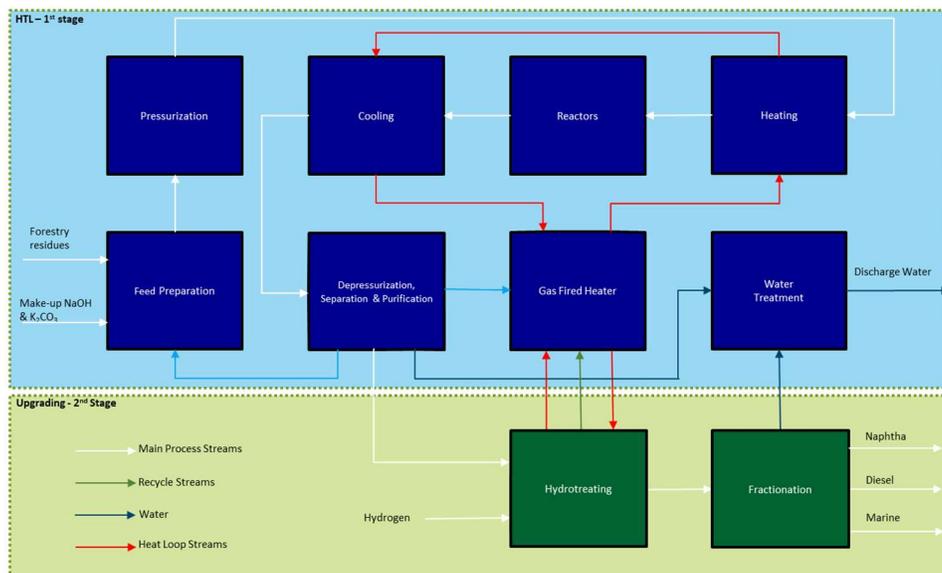


Table 1: Mass-, Energy- and elemental balances based on the average values presented in Figure 2. [15]

	Feed (Wood)	Biocrude	Off Gas	Water ^a	Total out
<i>Mass and Energy balance</i>					
Mass [wt.%]	100	45.3	41.2	13.8	100.3
Energy [%]	100	85.6	15.8	0	101.4
<i>Elemental balance</i>					
C [wt.%]	100	73.7	26.2	0	99.9
O [wt.%]	100	10.5	61.1	28.4	100.0
H [wt.%]	100	63.8	25.4	25.1	114.3

^aDetermined based on 100% oxygen balance

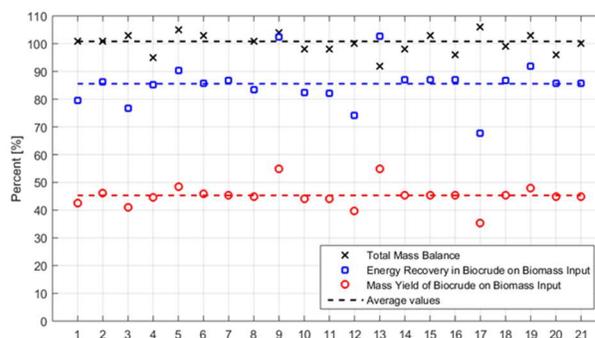


Figure 2: Key numbers on efficiency for steady state production of renewable crude oil. [15]

3. Upgrading pathways strategy to finish fuel oils and refinery integration

As with conventional fossil crude oils, Hydrofaction™ Renewable crude oil needs to be upgraded/refined before it can be used in transport fuel applications. As mentioned above, Hydrofaction™ Oil has distinct properties that need to be addressed during upgrading and they include high viscosity, high TAN, Oxygen content and low-sulphur content. These properties can lead to operational challenges such as:

- Reactors temperature control, due to high exotherms during biocrude's deoxygenation and stabilization.
- Upgraded oil recycling with potential clogging problems due to solubility reduction between upgraded oil and biocrude oil;
- Potential mass transfer issues due to decreased oil viscosity after oxygen and TAN reduction; and,
- Deactivation of commercial sulphided catalysts due to the low sulphur content of the biocrude (< 200 ppm); e.g. sulphur spiking is required to maintain the activity of many conventional sulphide CoMo and/or NiMo type catalysts [13] or non-sulphided catalysts must be used.

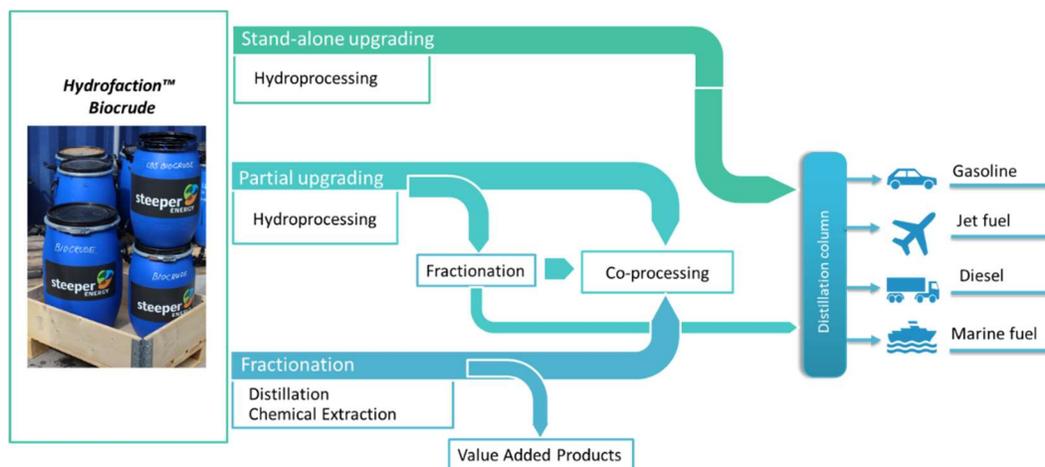
To address these issues, an extensive program for characterizing and upgrading the biocrude oil to “Drop in” transportation fuels or blend stocks has been performed using facilities at University

of Calgary and University of Alberta in Canada as well as at Aalborg University in Denmark. This program has been important to understand the characteristics of the oil, demonstrate its upgradability and market value. One of the aims of this upgrading program has been to employ different upgrading strategies including stand-alone and refinery co-processing in order to maximize the yield of renewable diesel that is on or close to specification (*Figure 3*), which allows for utilization of diesel infrastructure and engines established over the last 150 years.

Different upgrading strategies, catalysts, reactor configurations, temperatures, and pressures have been tested both in screening experiments and longer-term stability tests (>700 hours). Those tests have proven that the oil is upgradable if the upgrading process is modified to accommodate the biocrude distinct properties. For example, a modified catalyst bed configuration and the ability to remove water product after deoxygenation reactions are required. Key results are shown below in Figure 4-Figure 5 and Table 2Table 3 and further described in detail in [4], [12], [13], [18]–[22].

Co-processing studies have been successfully conducted at CanmetENERGY in Devon, a federal petroleum research lab under the Department of Natural Resources Canada. Tests were conducted using blends containing up to 10% of the distillates fraction of Hydrofaction™ biocrude with petroleum-based feedstocks in a continuous bench scale hydroprocessing unit for about 1400 hours under industrial relevant conditions. The results suggested that co-hydroprocessing of the biocrude with heavy fossil distillates is a suitable point for Hydrofaction™ renewable crude oil refinery integration.

Figure 3:
Hydrofaction™
Renewable crude
oil strategy



Biocrude’s upgrading results shown in Figure 4 and Table 2 were achieved by a combination of hydroprocessing stages using commercial sulphided catalysts. A significant reduction of boiling point and residue is obtained after partially upgrading the biocrude, i.e. the fraction from the initial boiling point (IBP) to 350 °C is more than doubled by the upgrading process, and the residue (BP > 550 °C) is reduced from 28% to 7.9%. Also, it is worth noting that the total acid number and oxygen content is reduced to below detection level for the fully upgraded oil, and the hydrogen to carbon ratio has been increased from 1.25 to 1.73. Table 3 shows the properties of the different fractions after distillation according to ASTM D2892 [23]. Figure 5 shows pictures of the biocrude and the fractions obtained after upgrading process.

Figure 4: Boiling point distribution curves determined by simulated distillation for renewable crude oil, partial upgraded crude oil, and upgraded oil.

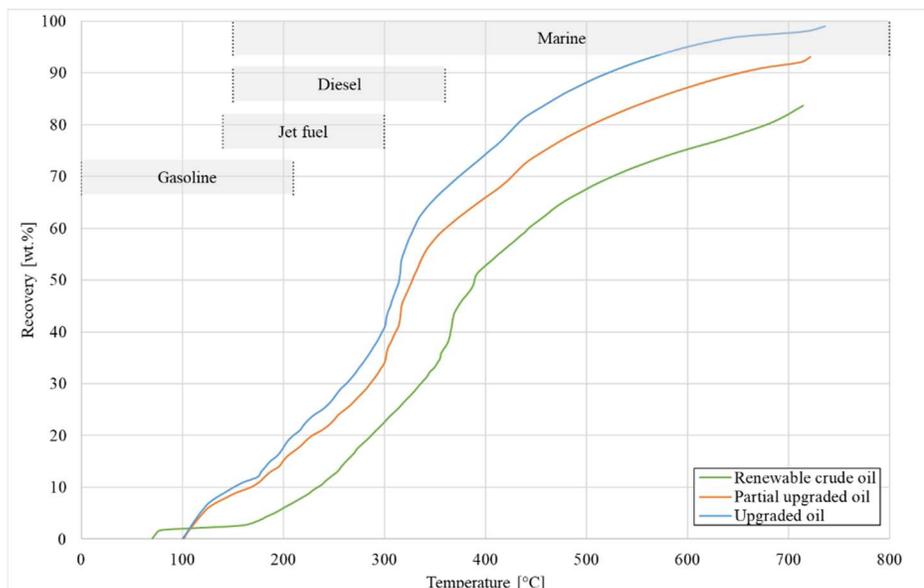


Table 2: Comparison of key properties for renewable crude oil, partially upgraded oil and upgraded oil.

	Renewable crude oil	Partial Upgraded Oil	Upgraded oil
Density at 15 °C [kg/m ³]	1051	926	903
Aromatic content [mol%]	-	21.1	17.7
TAN [mgKOH/g]	55.7	1.4	<0.1
HHV ^a [MJ/kg]	37.6	43.9	44.3
C, wt.% (daf)	81.4	87.4	87.3
H, wt.% (daf)	8.5	12	12.7
O, wt.% (by diff.)	10.1	0.6	0
H/C	1.25	1.64	1.73
IBP-350 °C distillate [%]	32	64	67
Residue > 550 °C	28.0	16.3	7.9

Partial upgraded oil obtained at 350-370 °C, Pressure: 100 bar, Commercial sulphide catalyst, 0.5 h⁻¹ space velocity (SV);
Upgraded oil obtained at 370 °C, Pressure 110 bar, Commercial sulphide catalyst, 0.3 h⁻¹ SV.



Figure 5: Pictures of crude oil and the clear fractions of the upgraded product.

As seen from Table 3 above, the Jet- and diesel fractions F2-F5 have H/C ratios in the range 1.73-2.02 and higher heating values of about 45 MJ/kg. These values are similar to those of conventional gasoline and diesel. It should also be noted that the 350+°C fraction has higher aromatic content which reflects the higher density and H/C ratio. About 7 wt.% of upgraded product ends up as potential gasoline blendstock. The gasoline fraction has been found to be compatible with fossil gasoline in any concentration, however, due to the low flash point, it has been found difficult to blend with other fossil fractions while complying to an existing standard.

Various diesel and marine fuel blends were physically produced from the Jet/diesel fuel fraction and the 350+°C fraction. The blend percentages were determined based on predictions from optimized blending models for compliance with the EN590 (2013) [24] ultra-low sulphur diesel standard and ISO 8271 (2012) [25] specifications for low sulphur marine fuel oil. A diesel blend was achieved comprising 30 vol.% renewable blendstock and complying with EN590 Ultra low sulphur diesel standard. The amount of renewable blendstock that can be added is currently limited to 30 vol.% due to high cetane number and density of the F2-F6 fractions, which is caused by the residual aromatic content. Upgrading work is ongoing to reduce aromatics and improve the

overall properties of the upgraded oil in order to push these blend limits. On the marine fuel side, a series of blending tests were performed utilizing the 350+ °C fraction of partially upgraded Hydrofaction™ Oil in a low sulphur RMG180 marine fuel formulation complying with ISO 8271 (2012). Physical blends were produced, and the compatibility and stability of the blends were evaluated using spot tests and microscope studies. An RMG 180 marine fuel blend comprising 62 vol.% of the 350+ °C fraction complying with the ISO 8271 (2012) standard for low sulphur RMG180 marine fuels was achieved. The results indicate the possibility of using the blends in existing engines without engine modifications.

Table 3: Properties of upgraded oil and distillation fractions [12]

	Upgraded Oil	F1	F2	F3	F4	F5	F6	F7
Temperature range [°C]	-	54-150	150-200	200-250	250-300	300-325	325-350	350+
Yield [wt.%]	-	7.1	10.2	9.1	12.0	17.8	10.8	31.6
Density at 15 °C [kg/m ³]	903	770	814	863	872	866	921	1033
HHV ^a [MJ/kg]	44.3	44.8	45.1	45.0	45.2	45.4	44.3	43.1
Aromatic content ^b [mol.%]	17.7	3.4	8.2	11.8	12.8	10.6	19.6	33.0
Flash point [°C]	37.5	<25	44.5	82	117	151	169	NA
CCI ^c	-	-	35	32	44	59	46	NA
S ^d [ppm]	30	54	19	23	10	15	34	NA
C [wt.%]	87.3	85.0	85.5	86.4	86.5	86.2	87.3	89.4
H [wt.%]	12.7	15.0	14.5	13.6	13.5	13.8	12.7	10.6
O ^e [wt.%]	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
H/C	1.73	2.10	2.02	1.87	1.86	1.91	1.73	1.42

^a dry basis; ^bby C13-NMR; ^c estimated from density and distillation data; ^d deviation: up to 50 ppm; ^e by difference. [4]

4. Life Cycle GHG emissions of Hydrofaction™

A life cycle evaluation of GHG emission reductions associated with Hydrofaction™ products has been performed in parallel with the present project. The scenario is based on a 2000 barrel-per-day (BPD) Hydrofaction™ plant consuming forestry residues and producing diesel and marine drop-in renewable fuels. Sankey diagrams for the overall mass- and energy flows for the 2000 BPD Hydrofaction™ plant assessed in the GHG emissions calculation are shown in Figure 6.

As shown in the Sankey diagrams below the gas products of Hydrofaction™ contain significant energy. Combustion of these gases (in steady state operations) provides sufficient heat for the requirements of the overall process. Nonetheless, an external LNG stream is included in the GHG analysis in order to cover start-up fuel requirements. On a net energy balance basis 71% of the energy input is recovered in the “drop-in” fuels.

The province of Alberta, Canada, is a suitable location of the Hydrofaction™ plant due to the accessibility to abundant forestry residue [6] and fuel supply and distribution infrastructure. i.e. extraction, refining, and distribution of oil and derived products. Details of the emission intensities used in the GHG analysis of the Hydrofaction™ plant are reported in [13]. The main assumptions of the analysis are as follows:

- The Hydrofaction™ plant uses forestry residues that under the baseline scenario would have been disposed in permanent wood waste stockpiles.
- The feedstock is transported from the harvesting site over 200 km on average;
- The Hydrofaction™ process gases are combusted to provide process heat
- The emission intensities of CH₄ and N₂O combustion products are equal to those from combustion of liquefied natural gas (LNG).
- LNG is used to cover any additional heat requirements related to process start-up.

- Standard grid electricity emission factor is used (Alberta average a per biofuel protocol)
- Heat, electricity and make-up HTL catalyst input requirements are based on the engineering studies for a 2000 BPD Hydrofaction™ plant.
- Upgrading catalysts are changed once every year
- The Hydrofaction™ make-up catalysts are transported over 1000 km on average by heavy truck
- Hydrogen consumption for upgrading is a conservative 4 wt.% (oil feed basis)
- The upgraded Hydrofaction™ oil is fractionated into 50 vol.% renewable drop-in diesel fuel and 50 vol.% drop-in marine fuel, which are used to displace petroleum diesel and heavy fuel oil, respectively.
- GHG emissions related to distribution and dispensing of the finished fuels is left out of the calculation as it is assumed to be the same in both baseline and project scenarios
- GHG emissions related to construction of the plant are assumed negligible over the lifetime of the plant.

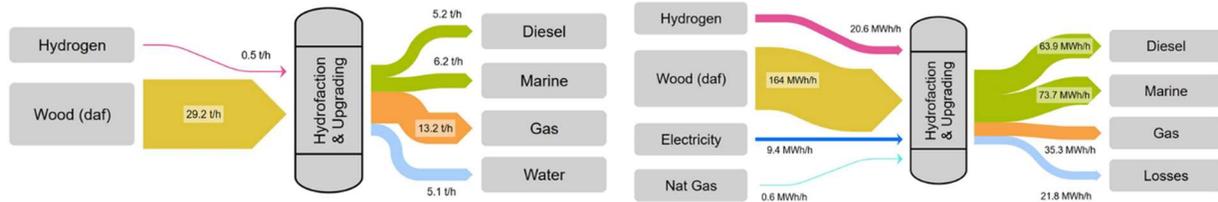


Figure 6: Sankey diagrams showing the mass- and energy balances for a 2000 BPD Hydrofaction™ plant on a dry ash free basis [13].

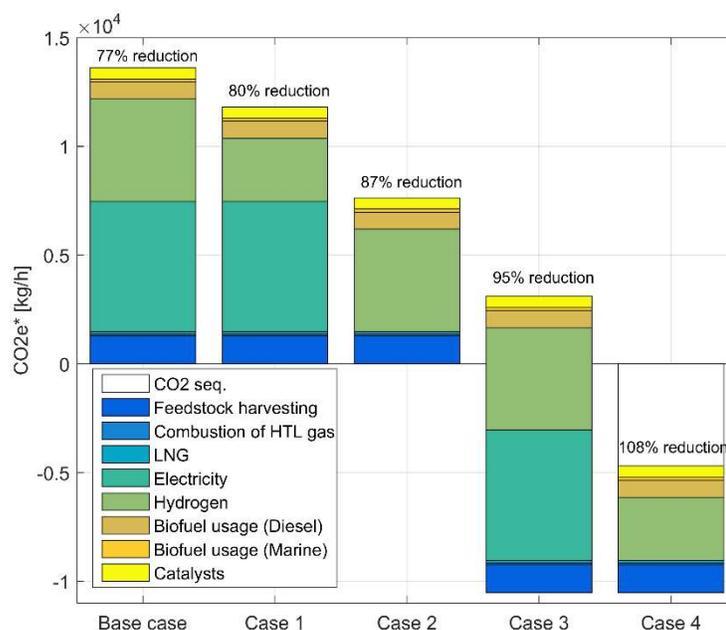
Different scenarios of GHG emissions were tested in the Life Cycle Assessment and compare base on the resulting emissions reduction (Figure 7). The baseline scenario is based on purchase of all hydrogen required for upgrading, methane emissions from decomposition of woody waste stockpiles (calculated based on the guidelines in the carbon offset emission factor handbook [26]), use of standard grid electricity (coal-intensive in Alberta) and where the biogenic CO₂ from the HTL gaseous products is all emitted. As present in Figure 7, the major contributions to GHG emissions in the base case scenario are related to the purchased electricity and hydrogen, covering 44% and 35% of the total emissions respectively. Based on that, the following four additional scenarios (as presented in [13]) were tested in the LCA (more detail available in [13]):

- Case 1:** Hydrogen produced in-situ during 1st stage Hydrofaction™ is used during upgrading of the renewable crude oil to lower the amount of hydrogen that need to be purchased. No additional LNG need to be purchased, because there is sufficient heating value in the remaining gaseous products after hydrogen separation. Additional electricity demand is not accounted for.
- Case 2:** Renewable grid electricity is used to reduce the emissions intensity of electricity, but at the expense of a higher cost.
- Case 3:** All the biogenic CO₂ from the HTL gaseous products is extracted (before combusting the gaseous products with air) and sequestered. Additional electricity demand is not accounted for.
- Case 4:** Include all of the above.

The effect that each of these cases have on the emissions savings of the Hydrofaction™ project base cases are plotted in Figure 6 below. The effect of using in-situ produced hydrogen in Case 1 appears minor, but this approach may have major economic benefits as the purchase and logistical complications of bringing external hydrogen can incur significant costs to the project. In

Case 2 the effect of using of renewable electricity instead of standard grid electricity appears to have a major effect on the GHG emissions as power generation in Alberta is relatively carbon-intensive due to high abundance of coal-fired power plants in the Province. The economic considerations of sourcing renewable electricity depend on carbon pricing and regulations. Case 3 shows that an additional 19% in GHG emission savings is achieved when implementing recovery and capture of CO₂ from the Hydrofaction™ process gas; (Steeper Energy has own IP in this CO₂ recovery add-on to Hydrofaction™ [27], [28]). Case 4 adds all the above modifications to show total GHG reductions of 108% compared to base case, indicating that Hydrofaction™ can deliver CO₂-negative projects.

Figure 7: GHG emissions savings of different Hydrofaction™ project cases [13]



Conclusion

Steeper Energy, by means of Hydrofaction™ technology and subsequent biocrude upgrading and refining, aims to the production of fuels for long-haul transport (i.e. heavy trucks, maritime and aviation). Significant upgrading efforts over the past two years included successfully operating four continuous pilot plants in parallel in different process configurations in campaigns of more than 700 h each without catalysts deactivation. The physicochemical properties of the Hydrofaction™ renewable crude oil were significantly improved during hydroprocessing using commercial sulphided catalysts. Additionally, successful blending of diesel fraction (up to 30% vol.%) with fossil diesel and the fraction with a boiling point higher than 350 °C (up to 62 vol.%) with Marine fuel oils (RMG 180) were obtained. These blends comply with the specification of Diesel EN590 (2013) for ultra-low sulfur diesel and ISO 8271 (2012) for Marine fuel. Mass and energy balances for a 2000 BPD Hydrofaction™ plant indicate that 1 tonne of forestry residues can be converted to more than 400 L of renewable diesel and marine fuels with an energy recovery of 71% and GHG emission reduction of 77%-108%, reflecting an energy and resource-efficient technology for the production of renewable fuels for long-haul transportation.

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