IEA Bioenergy

Task 34

Direct Thermochemical Liquefaction

- Welcome New Zealand to Task 34
- Welcome Back Canada to Task 34

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Welcome to Task 34

2016: Expanded Countries, Expanded Emphasis

On behalf of myself and my colleagues from Canada, Finland, Germany, the Netherlands, New Zealand and Sweden:

Welcome to the first year of the new Triennium of IEA Bioenergy Task 34.

Please take a moment to meet the National Team Leads on page 2, and learn more about the Task on page 20. This issue contains an excellent mix of articles on both pyrolysis and solvo-liquefaction.

The Task has undergone a series of changes this year: Welcoming new countries and new colleagues, and saying farewell to good friends and pioneers in this field. One of the most impactful changes is our expanded focus on Direct Thermochemical Liquefaction.

The objective of Task 34 is to facilitate commercialization of liquid fuels from biomass through direct thermochemical liquefaction, particularly fast pyrolysis and solvo-thermal processing. Our goal is to contribute to the resolution of critical technical areas and disseminating relevant information particularly to industry and policy makers.

During 2016, two statements by senior researchers in biomass liquefaction have influenced my thinking. They are paraphrased in the boxes below:

> “It important to understand the difference (and similarities) between liquefaction oils and pyrolysis oils.”

Bio-oils from pyrolysis and bio-crudes from solvent-liquefaction oils have many qualitative similarities, yet many quantitative differences. There is an expanded need for analysis of the bio-crudes. There is a need to apply the knowledge around bio-oils to advance bio-crude applications. An equal need is to learn from the characteristics of bio-crudes to advance the quality of bio-oils. A goal of this Task is to elucidate these similarities and differences.

> “Some transfer of knowledge between teams occurs best when the lab-level researchers who work with equipment talk together.”

On a tour this year of a biomass pilot plant, I observed two researchers meet for the first time and start to share the difficult lessons they had learned about equipment

(Continued on page 2)

Welcome…continued

configuration in solvent liquefaction. It was not information found in the typical journal article, but in the trials and choices in applied laboratories around the world. That valuable conversation will enable both those scientists and their teams to have more successful research in the future.

Ultimately, I believe that the challenges we face for the adoption of renewable biomass energy technologies are larger than the work of any one person, technology, organization, or country. It will require significant international and personal collaborations from all of us in order to work together to solve these challenges and further the impact of renewable energy in all of our countries. Thank you for being part of this community.

As we go forward, I commit to you to look for opportunities where we can each learn more and teach others about the lessons we have learned in pyrolysis, solvo-liquefaction, upgrading, and end use, such that we might advance these technologies together.

I hope that you, as a member of this research community, will also look for these opportunities and encourage others to do the same.

As you look through the articles in this newsletter, please email or call the contact the author with whom you have similar research interests and challenges to find opportunities where you can collaborate. Also, please contact your national team lead if you have not already done so.

Our next task meeting is in Rotorua, New Zealand in November.

I wish you successful research in 2016.

Alan Zacher
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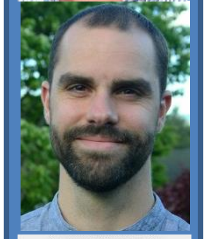

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European standard for fast pyrolysis bio-oil for boilers almost finalised

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Table 1: Overview of standardisation committees under CEN and ISO for solid, liquid and gaseous biofuels.

Committee	Name	Remarks
CEN/TC 335	Solid biofuels	Standardisation work has finished and new standards, which supersede these standards, will be published under ISO/TC 238.
ISO/TC 238	Solid biofuels	Standardization of terminology, specifications and classes, quality assurance, sampling and sample preparation and test methods in the field of raw and processed materials originating from arboriculture, agriculture, aquaculture, horticulture and forestry to be used as a source for solid biofuels. Vienna agreement[1] to be followed and standards will be published in Europe as EN ISO standards.
CEN/TC 019	Petroleum products, lubricants and related products	WG 41 to develop quality specifications for pyrolysis products for boilers and stationary internal combustion engines
CEN/TC 343	Solid recovered fuels	Solid recovered fuels prepared from non-hazardous waste. Work is finished and new standards will be published under ISO/TC 300.
ISO/TC 300	Solid recovered fuels	Elaboration of standards and other deliverables on solid recovered fuels prepared from non-hazardous waste to be utilised for energy recovery in waste incineration or co-incineration plants or in industrial processes (like cement manufacturing), excluding fuels that are included in the scope of ISO/TC 238.
CEN/TC 383	Sustainably produced biomass for energy applications	Sustainability principles, criteria and indicators including their verification and auditing schemes, for as a minimum, but not restricted to, biomass for energy applications. This includes GHG emission and fossil fuel balances, biodiversity, environmental, economic and social aspects and indirect effects within each of the aspect, include only sustainability of biofuels and bioliquids for energy applications.
ISO/TC 248	Sustainability criteria for bioenergy	Sustainability criteria for production, supply chain and application of bioenergy. The work has finished and 4 standards published.
ISO/TC 255	Biogas	Standardization in the field of biogas
CEN/TC 408	Natural gas and biomethane for use in transport and biomethane for injection in the natural gas grid	Standardization of specifications for natural gas and biomethane as vehicle fuel and of biomethane for injection in the natural gas grid, including any necessary related methods of analysis and testing. Production process, source and the origin of the source are excluded.

[1] Vienna agreement signed in 1991 was drawn up with the aim of preventing duplication of effort and reducing time when preparing standards. As a result, new standards projects are jointly planned between CEN and ISO. ISO standards will be published in Europe as EN ISO standards and voting of different phase will carried out parallel.

Standardisation of biomass fuel is active in Europe and internationally. Several committees are working to develop standards for liquid, gaseous and solid biomass fuels. Working group 41 under CEN Technical Committee 19 started to draft European standard for FPBO for boiler use in 2014. The standard EN 16900 is almost finalised and planned to be published in the beginning of year 2017. Working group 41 is currently also drafting a technical report of FPBO (CEN/TR) for stationary internal combustion engines. The original plan was to draft technical specification (CEN/TS), but because FPBO is not yet fully commercialized for stationary internal combustion (IC) engines and there is neither enough data on critical properties for engine use nor the long-duration tests in IC-engines, WG41 proposed to TC19 to publish it as CEN/TR.

Standardisation of biomass related fuels

Standardisation of biomass related fuels is active in Europe and internationally. CEN and ISO technical committees are listed below. Fast pyrolysis bio-oil (FPBO) is developed under CEN/TC 19. Raw materials for FPBO can be specified according to ISO 17225-1:2014, which is published under ISO/TC 238.

(Continued on page 4)

European standard for fast pyrolysis...continued

European standard for fast pyrolysis bio-oil (EN 16900)

Biomass fast pyrolysis bio-oil (FPBO) is entering into the market in Europe. Currently, the first-of-its kind commercial installations for fuel production are in operation in Finland (Fortum) and in the Netherlands (Empyro). The European Standard (EN 16900) specifies requirements and test methods for FPBOs for boilers used at industrial scale (>1 MW thermal capacity), not for domestic use. Two different grades are specified. In addition to the quality requirements and test methods for FPBOs, further instructions on storage, sampling, and materials compatibility are given. Test methods include also modification for the methods recommended for FPBO. The precision data from inter-laboratory test survey (ILS) are also given in the standard. Full report of ILS results will be published as a report by the end of year 2016.

Figure 1: Moisture analysis is one of the important properties of bio-oil. Photo: VTT

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For emission and burner dependent requirements, options are given to allow grades to be set locally or chosen by the user. The options are two grades, of which Grade 1 requires more flue gas treatment than Grade 2. When tested by the methods indicated in Table 3, fast pyrolysis bio-oils shall be in accordance with the maximum limit for the Grade applicable. The test methods listed have been assessed for application in boiler use.

(Continued on page 5)

Table 2: Property requirements and test methods for fast pyrolysis bio-oils for boiler use (FprEN 16900, June 2016)[*]

Property	Unit	Test Method
Net calorific value as received[a]	MJ/kg	DIN 51900–3
Water content, on wet basis	%(*m/m*)	ASTM E203
pH		ASTM E70
Density at 15 °C	kg/dm^3	EN ISO 12185
Pour point	°C	ISO 3016
Nitrogen content	% (*m/m*) (d.b.[b]).	ASTM D5291

[a] Net calorific value as received is calculated from the gross calorific value according to DIN 51900-1.

[b] d.b. is on dry basis.

[*]Threshold values of Table are not published because of CEN copyright reasons.

Emission and burner dependent property requirements and test methods for fast pyrolysis bio oil for boiler use (FprEN 16900) for Grade 1 and Grade 2[*].

Property	Test method	Unit
Kinematic viscosity at 40 °C	EN ISO 3104	mm^2/s
Sulfur content	EN ISO 20846	%(*m/m*), d.b. [a]
Solids content	ASTM D7579	%(*m/m*)
Ash content	EN ISO 6245	%(*m/m*), d.b. [a]
Na, K, Ca, Mg	EN 16476	%(*m/m*) d.b. [a]

[a] d.b. on dry basis

[*]Threshold values for Grade 1 and Grade 2 in Table are not published because of CEN copyright reasons.

European standard for fast pyrolysis…continued

Figure 2: FPBO fuelled process steam boiler at FrieslandCampina in Borculo, the Netherlands. Up to 4 t/hr of FPBO is co-fired with natural gas". Picture Empyro/FrieslandCampina

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Direct liquefaction of lignin and lignocellulose by catalytic hydrogenolysis

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Figure 1: Micro Autoclaves 25 ml, stainless steel, for temperatures up to 500 °C and pressures up to 300 bar
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As part of an ongoing project Institute of Catalysis Research and Technology (IKFT) at Karlsruhe Institute of Technology (KIT), Germany, the direct liquefaction of lignin and lignin-rich biomass under conditions of direct coal liquefaction (DCL) is investigated. The objective is to use the similarities in structure and elemental composition of lignin and lignite transferring the well-known Bergius process onto lignocellulose conversion. By adapting this process it is possible to access bio-based aromatic components for use in chemical industry as well as for liquid fuels or additives.

Learning from Bergius Process

In the Bergius process of direct coal liquefaction [1] fragments of low molecular weight are cleaved from coal structure in the presence of a catalyst, a hydroaromatic solvent and hydrogen gas. These fragments are capped by hydrogen and therefore stabilized before polymerization by recombination reactions may occur. Thus the supply of hydrogen is of major importance for the liquefaction process [2, 3].

The appropriate choice of the solvent plays an important role. It may act as an additional source of hydrogen (hydrogen donor) or it can facilitate the transfer of hydrogen from gas phase to the reactive sites (hydrogen shuttle). The most effective solvents are partially hydrogenated polycyclic aromatic hydrocarbons, which can act as both a hydrogen donor and a hydrogen shuttle [4]. Since Tetralin matches both the requirements it was chosen for the investigation.

As catalysts, iron and molybdenum in sulfidic or oxidic form have been proved to be effective catalysts for DCL. During the liquefaction process char and mineral constituents accumulate in the solid residue causing deactivation of the catalyst and hampering its recovery. Therefore it became apparent that it is economically expedient to use cheap iron salts or even suitable waste products from industry like red mud as a one-way catalyst.

Preliminary Screening

In a first step screening of different types of catalysts and biomass was performed using unstirred micro autoclaves with an inner volume of 25 ml (Fig. 1). The experiments were carried out at a reaction temperature of 500 °C and a hydrogen pressure in the cold autoclave of 100 bar, resulting in reaction pressures of up to 300 bar. As feedstock three commercial lignosulfonates (Waffex Ca, Borrespers AM, Lignex MgF) and one Kraft lignin (Indulin AT) were chosen. Both molybdic acid (4%) and an iron/sulfur-catalysts on carbon carrier (10% ≙ 2% catalytically active Fe-S-component) were used as a catalyst.

Figure 2 shows the product yield obtained from the experiments. Liquid product yields for lignin are in the range of 35 to 60% while the gas yields reach 35 to 50%. The solid residue is strongly depending on the ash content of the lignin used. Borrespers

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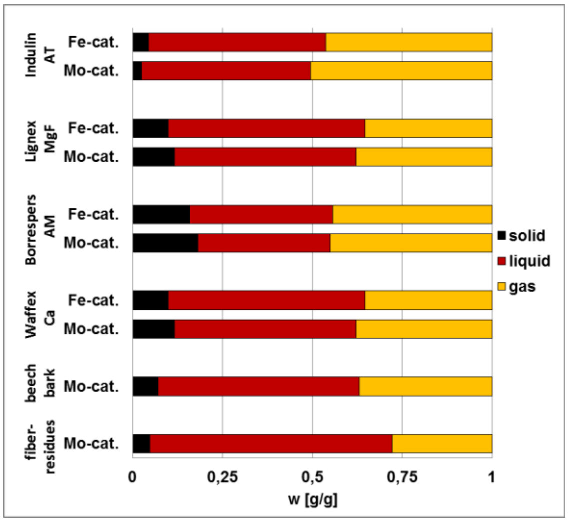

Figure 2: Results of screening four different types of lignin, beech bark, and fiber residues from pulp and paper industry with Mo- and Fe/S-catalyst at 500 °C, 300 bar for 1 h residence time (solid with ash and catalyst residue)

Direct liquefaction of lignin and lignocellulose ...continued

Figure 3: Stirred Tank Reactor 2000 ml, for temperatures up to 500 °C and pressures up to 300 bar

AM with the highest ash content shows the highest amount of solid residue while the experiments with low ash lignin Indulin AT leave only small amounts of solid residue. In these experiments no significant amount of aqueous phase was received. Additional experiments with milled beech bark and fiber residues from pulp and paper industry (Fig. 2) show comparable results.

Since with the cheaper iron/sulfur-catalyst similar yields were obtained as for the molybdic acid catalyst, it was chosen for the further investigations.

Mini plant

Next, experiments were conducted in a new experimental setup utilizing a 2000 ml stirred tank reactor (Fig. 3). This allows providing higher amounts of product for adequate analytical characterizations and testing. Three parameters were selected to be investigated - reaction temperature, catalyst concentration, and addition of elemental sulfur for promotion of the catalyst.

Variation of the temperature allows controlling product distribution and quality. At higher temperatures the amount of gas increases at the cost of liquid product yields (Fig. 4). This is a result of thermal cleavage of alkylic and hetero-functional sidechains of the aromatic rings from the gained oil product. In return the liquid products have lower boiling ranges and viscosities, meanwhile at lower temperatures, when the fragmentation of the side chains is less; there are higher yields of liquid products of higher boiling ranges and viscosities. The best results were achieved at a reaction temperature of 435 °C.

By varying the catalyst concentration it became obvious that the catalyst concentrations used in the experiments were higher than necessary (Fig. 5). There were no better results for higher catalyst concentrations. The results indicate that the catalyst is not involved in the cleavage of lignin structure itself, but in the supply of hydrogen. Using 10% of the iron-sulfur-catalyst resulted in the best yields regarding liquid products. Even lower amounts of catalyst may lead to the same results.

Additional supply of elemental sulfur did not show any effect on the reaction. For the use of technical lignin from common pulping processes this may be due to the fact that the lignin itself supplies enough sulfur for the formation of the catalytic active species. The addition of sulfur can be more interesting for the liquefaction biomass with low sulfur content.

To rule out an influence of the active carbon carrier - the lignin in another test was prepared by direct impregnating with the iron/sulfur catalyst. It was also supposed, that the so achieved better dispersion of the catalyst can improve the yields. The results can be seen in Fig. 6 in comparison to an experiment with catalyst on active carbon. The yields of gaseous and liquid products are almost identical. Only the solid residue increased noticeably. So even if the catalytic effects did not affect the product yields the impregnation method can be an improvement to the process because the additional carbon material is evaded lowering the undesired amount of solids.

After all the obtained results show that liquefaction of lignin by catalytic hydrogenolysis under conditions of direct coal liquefaction is possible to achieve high yields of liquid and gaseous products.

For the used technical lignin yields of up to 80% of liquid product with a low amount of solid residue can be achieved. The gained oil phase consists mainly of aromatic monomers without hetero-atomic functional groups. This indicates that the ether and methoxy groups dominating the lignin structure were almost entirely removed by

(Continued on page 8)

Direct liquefaction of lignin and lignocellulose …continued

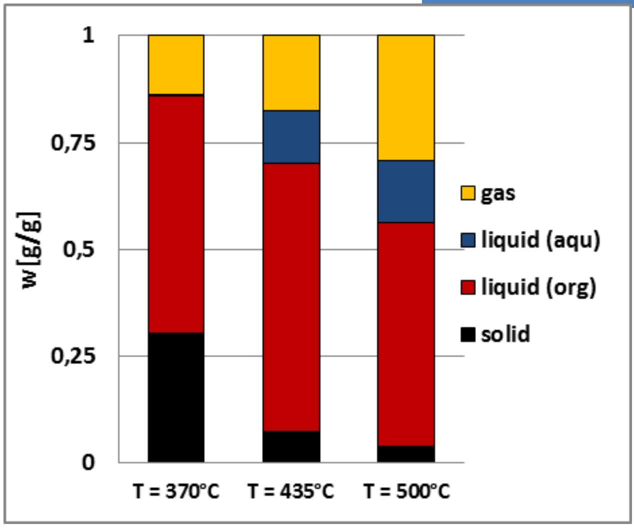

Figure 4: Mass balance for liquefaction of Indulin AT with Fe/S-catalyst at different temperatures

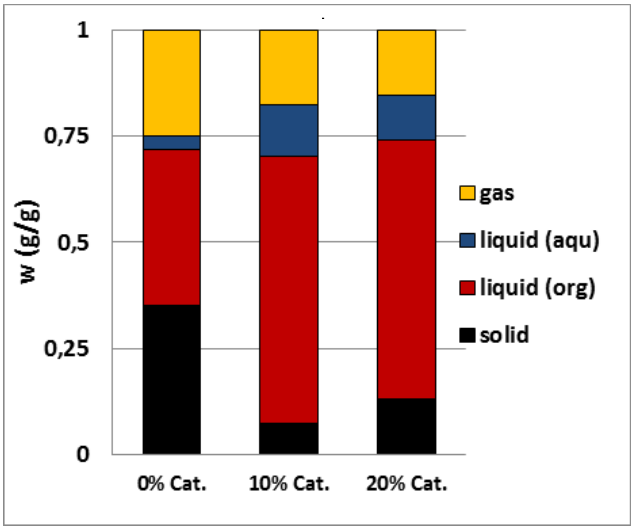

Figure 5: Results for liquefaction of Indulin AT at 435 °C at different concentrations of Fe/S-catalyst

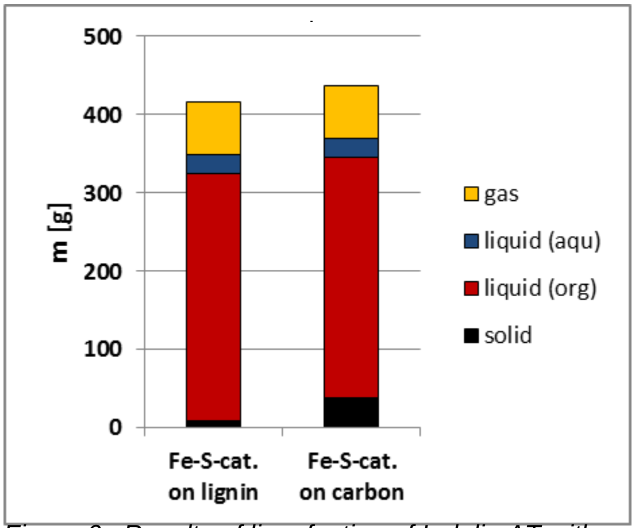

Figure 6: Results of liquefaction of Indulin AT with catalyst impregnated lignin and active carbon fixed catalyst on

hydrogenation under formation of water, CO, and CO2. Reaction water can be easily be removed by phase separation. The gaseous product containing more than 80% of hydrocarbons is also interesting for utilization.

Both molybdic acid and iron/sulfur catalysts show comparable results, but since a recovery of the catalyst from the solid product seems to be complicated, the iron based catalyst is more suitable for a possible industrial application because of their lower price. Impregnating the iron/sulfur catalyst onto the lignin proved to be the best method, because the solid residues can be reduced to a minimum.

The work was carried out within a project funded by Fachagentur für Nachwachsende Rohstoffe as the central coordinating institution for research, development and demonstration projects in the field of renewable resources. The PhD work is carried out in the thermochemical biomass conversion division at IKFT (https://www.ikft.kit.edu/237.php) of IKFT.

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The HTU® Process for the Hydrothermal Conversion of Biomass

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Figure 1: HTU® Pilot Plant (At the TNO site in Apeldoorn)

In the period 1997-2008 the HTU® Process was developed for the energy-efficient hydrothermal conversion of biomass residues to a liquid biocrude with a high calorific content. The development has been carried out at a 100 kg/hour pilot scale (see Figure 1) in three 2-3 year campaigns with several industrial partners (Shell, Stork Engineering and Construction, Total, HVC) and TNO.

The process is unique in its ability to process a wide range of cheap (wet) biomass residues, amongst which household waste, pig manure and digestates from anaerobic digestion. Furthermore, it is cost effective, energy efficient, scalable to a large scale for Biorefinery applications and is capable of a dedicated or simultaneous production of biodiesel, aviation fuel, chemical feedstock for ethylene/propylene crackers, biocoal for power stations (which do not need additional investments) and even cheap liquid CO2.

The HTU® process heats the feedstock in liquid water to temperatures between 300 and 350^{o}C, pressures of 100-180 bar and a residence time ranging from 5 to 20 minutes. At these conditions up to 85% of the oxygen is removed from the biomass. It ends up in about equal proportions in carbon dioxide and water. The thermal efficiency is 75-80% (percentage of feed heating value ending up in biocrude).
In chemical terms the key to biomass liquefaction is the removal of oxygen. Biomass contains typically 40-45%w (dry and ash free basis) of oxygen. Oxygen removal increases the heating value and it leads to a product with more hydrocarbon-like properties, ultimately causing it to be immiscible with water.

A very large variety of feedstocks has been tested in HTU® pilot plant and autoclave experiments. They include

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The HTU® Process...continued

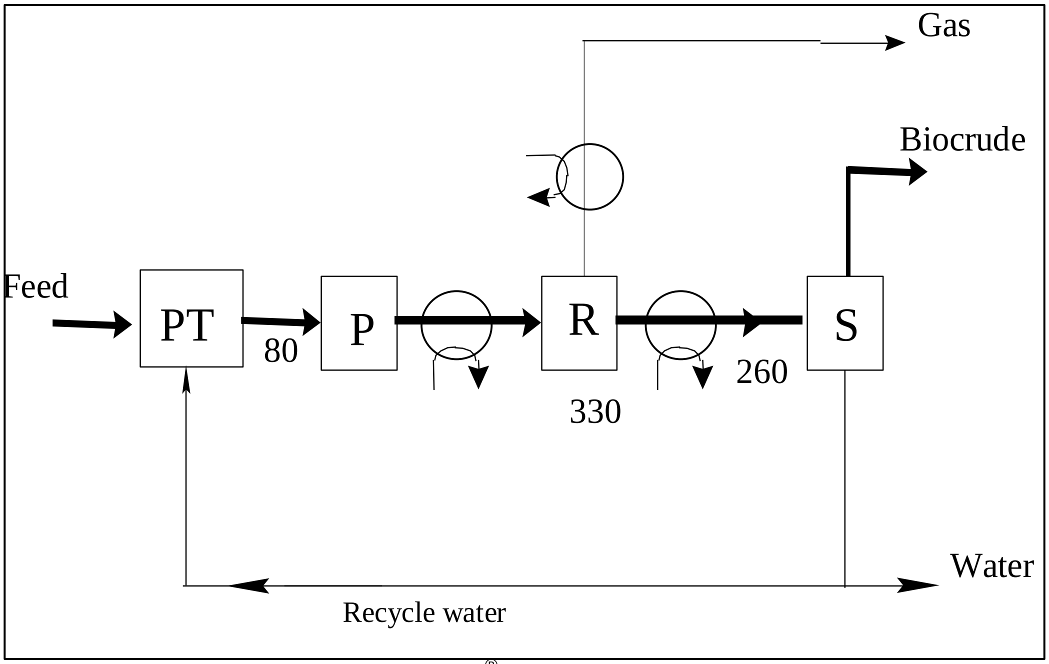

Figure 2: Simplified scheme of the HTU® process
PT = pretreatment, recycle water only if water content of feedstock is low.
P = pump; R =reactor; S =separator; temperatures in °C

wood from energy plantations, waste streams from sugar, potato and other food industries, grass, olive waste, organic domestic waste and fractions thereof, peat, digestate from anaerobic digestion, frying fat, slaughterhouse waste and pig manure. Figure 2 presents a simplified process scheme.

The biocrude can be separated into a light and a heavy biocrude by e.g. solvent extraction. The light biocrude is minerals-free. It can be upgraded by conventional hydrodeoxygenation (HDO) and hydrocracking to produce a gasoil and other hydrocarbon fractions, such as aviation fuel and naphtha as a chemical feedstock. Experimental evidence shows that a high-quality diesel fuel can be produced.
The heavy biocrude can be combusted, e.g. in a coal-fired power station.

The process is ready for the first commercial application at a scale of 25,000 tons of biomass (dry basis)/a. This has been confirmed in a Technical Due Diligence Study carried out in 2012 under supervision of a well-known international Engineering Contractor. After the first commercial demonstration there are substantial prospects for the process to utilize a significant part of the 5 billion tons/a of worldwide (wet) biomass residues.
Primary application areas are those that combine abundant resource availability and existing collection logistics with a disposal problem. Examples are municipal and domestic waste, forestry, palm oil residues, bagasse from sugar cane, olive oil extraction residues and pig manure. A prime candidate for initial application is the processing of municipal and domestic waste. The current large-scale burning is expensive and inefficient. It can be replaced by already available technology for separation of its valuable components, leaving a wet organic residue that is excellently suitable for HTU®.

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Renewable Residential Heating with Fast Pyrolysis Bio-oil: Residue2Heat

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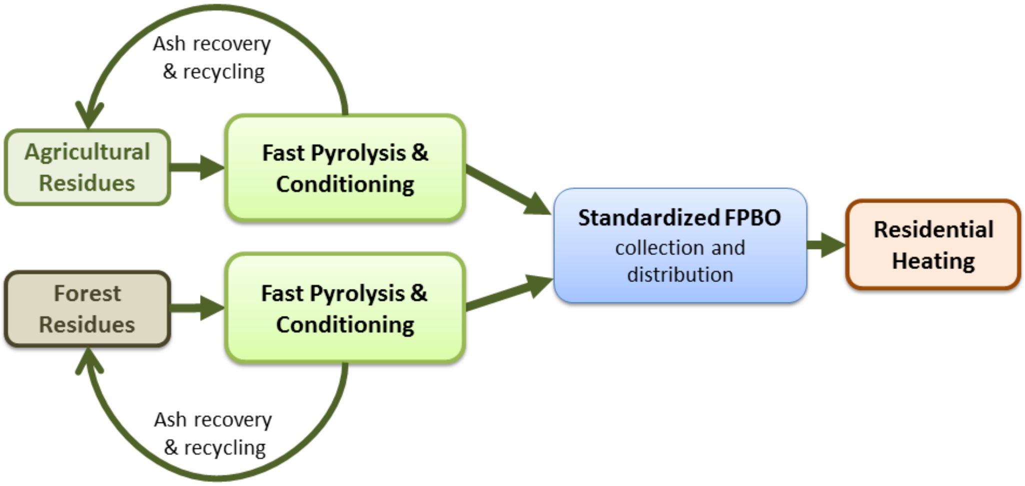

Figure 1: The overall concept of the EU research project "Residue2Heat" focusses on the development of a standardized pyrolysis oil from biomass residues, which can be used as fuel in the residential heating market. Graphics: OWI.

Residue2Heat is a new H2020 Research and Innovation Action initiated by 9 partners from 5 countries which aims at developing a concept for renewable residential heating using fast pyrolysis bio-oil. Alternative sustainable fuels for residential heating are needed in the household sector to reduce greenhouse gas emissions. There is a particular need to improve efficiency of low emission biomass heating systems while widening the feedstock base. This means that new flexible and robust residential-scale low emission boilers for heat applications need to be developed using a wider range of sustainable feedstock with its high ash contents.

Figure 1: The overall concept of the EU research project "Residue2Heat" focusses on the development of a standardized pyrolysis oil from biomass residues, which can be used as fuel in the residential heating market. Graphics: OWI

The overall concept of Residue2Heat is to use various streams of biomass waste for residential heat generation (Figure 1). The aim is, by means of a liquid fuel produced in a sustainable manner from agricultural and forestry biomass residues to considerably reduce the CO2 emissions in the heating market compared to fossil fuels.

This 2nd generation bio-fuel is being produced employing a fast pyrolysis process in which organic material is heated in the absence of oxygen to about 500 °C within a few seconds. Under these conditions organic and aqueous vapours, pyrolysis gas and charcoal arise. The vapours are rapidly cooled and condensed into a highly viscous liquid, so-called pyrolysis oil or

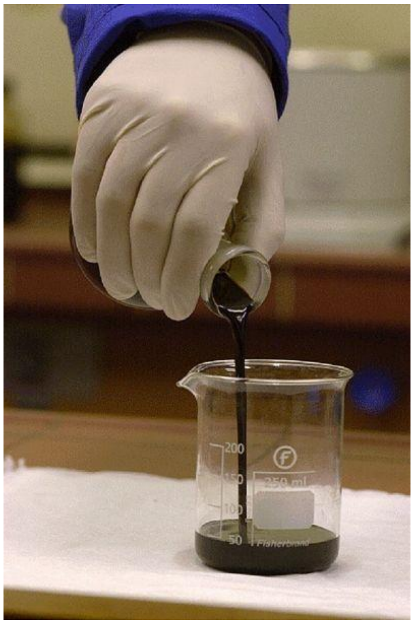

Figure 2: Pyrolysis sample [Copyright BTG Biomass Technology Group BV]

(Continued on page 12)

Residue2Heat...continued

Fast Pyrolysis Bio-Oil (FPBO). The FPBO has about 70% of the energy content of the biomass and about half of the heating value of a conventional fuel oil. An international consortium of research institutions and SMEs will examine the standardization and the use of FPBO in the heating market and optimize a condensing heating system for the use of this new fuel. The fuel is to be used for efficient heat generation in residential heating systems in the power range from 20 to 200 kW. Additionally, the valuable by-products (charcoal (10-15%) and low calorific gases (15-20%)) can be recycled to generate energy by combustion (Fig. 1), resulting in the production of ashes that contain the majority of the minerals and salts originally present in the feedstock and afterwards in the charcoal. In this context, the consortium also takes care of all the aspects related to the biological analyses of the ashes produced within the project and in particular of their microbiological properties. This relates to toxicity issues and effects on soils and plants, considering nutritive as well as inhibitory effects on the soil microbiota. Possible treatment or pre-treatment strategies will also be investigated, for example, the suitability for the preparation of ash-based composts to be used as fertilizer and/or soil conditioner. Also, the legal issues concerning the return of ashes to agricultural forestall, and horticultural land will be addressed at a European level.

The first challenge in this project is to produce FPBO despite its wide range of possible biogenic raw materials with a consistently high quality and highly standardized physico-chemical properties. Hence, FPBO conditioning is required and includes the removal of solids, extractives and/or water, and catalytic and non-catalytic alcohol upgrading, bio-oil blending, and their combinations. In addition, also increase in pH and decrease in TAN (total acid number) by alcohol upgrading is on focus. By using the FPBO route the high ash content in the feedstock can be recovered during the production of FPBO, leading to low ash emissions by the residential heating system at the end-user.

The second central challenge is the technical adaptation of a highly efficient condensing heating system for the use of FPBO, since the FPBO properties differ from those of conventional fuels. FPBO is presently entering into the heating oil market to replace fossil heavy and light fuel oils in stationary large scale boilers and furnaces. However, the development of an efficient small scale FPBO boiler requires a better understanding of FPBO combustion and spray parameters necessary for controlling and improving the system. In Residue2Heat the focus of development in particular is the mixture preparation, which is responsible for an efficient and low-emission operation of the burner. The applied burner concept is based on a so called blue flame type leading to low emissions compared to standard yellow flame type burners.
Finally, within the project market studies are conducted which assess the potential of this new fuel and the modified heating system and provide further knowledge for a successful market launch. The long-term goal of "Residue2Heat" is to produce FPBO on the basis of

Figure 3: Pyrolysis transport.
Copyright BTG Biomass Technology Group BV & Empyro BV

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Residue2Heat…continued

Residue2Heat

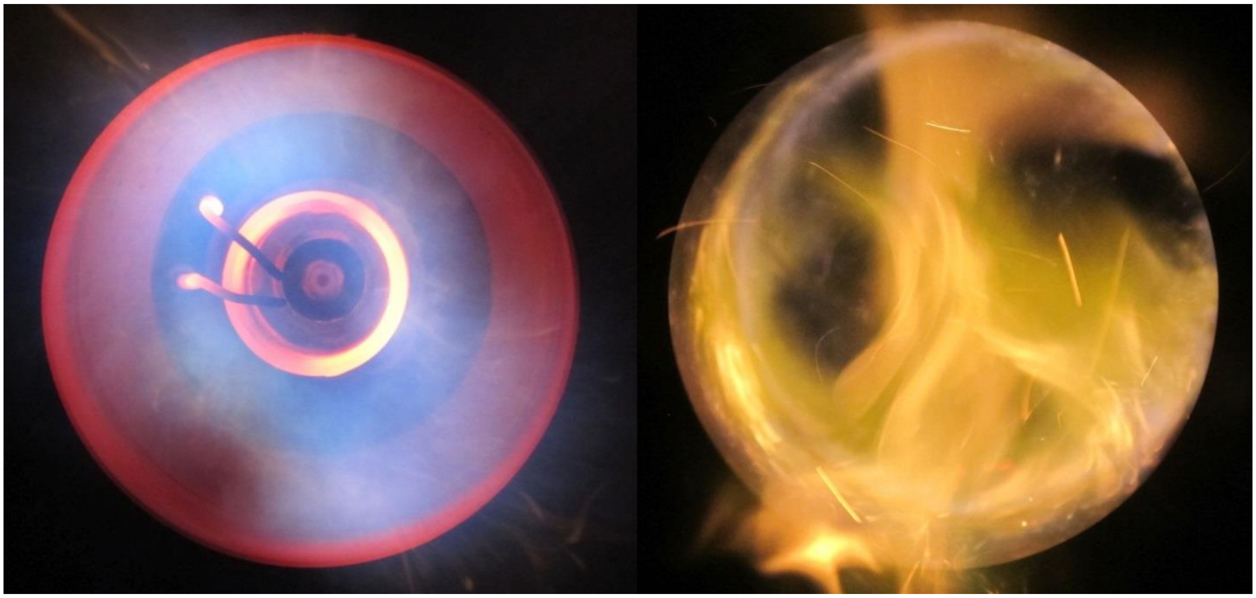
Figure 4: Left burner operated with heating oil, on the right the same burner without modifications operated with 50% Pyrolysis oil. Clearly visible is the less optimal combustion (yellow flames), leading to higher emissions. One of the challenging tasks in the Residue2Heat project will be the development of a highly efficient combustor with low emissions. [Copyright OWI Oel-Waerme-Institut GmbH]

agricultural and forestry biomass residues, which neither can be used for food or feed production nor leads to indirect land use change (ILUC). The conceptual approach aims to obtain local biomass, convert it into FPBO in relatively small production facilities with a processing capacity from 20,000 to 40,000 tonnes of biomass per year and distribute the fuel locally to end-users for residential heating.

The Residue2Heat project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No. 654650

Horizon 2020
European Union funding
for Research & Innovation

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More information is available at:
www.residue2heat.eu

About the Residue2Heat-consortium

The EU research project "Residue2Heat" combines the development of production technologies for the production of renewable fuels with the development of heating systems for the residential heating market. The project consortium is made up of three universities, three research institutes and three small and medium businesses from five different countries.

- *RWTH Aachen University (Coordinator, Germany)*
- *OWI Oel-Waerme-Institut GmbH (technical coordinator, Germany)*
- *Biomass Technology Group B.V. (The Netherlands)*
- *VTT Technical Research Centre of Finland Ltd. (Finland)*
- *MEKU Energie Systeme GmbH & Co. KG (Germany)*
- *Istituto Motori, Consiglio Nazionale delle Ricerche (Italy)*
- *PTM Politecnico di Milano (Italy)*
- *BTG BioLiquids B.V. (The Netherlands)*
  - *University of Innsbruck, Institute of Microbiology (Austria)*

PYRENA: PYROLYSIS EQUIPMENT FOR NEW APPROACHES IN CIRCULATING FLUIDISED BED CATALYTIC PYROLYSIS FOR BETTER BIO-OIL AS PRECURSOR FOR FUELS AND CHEMICALS*

Dr. Paul J. de Wild
Energy research Centre of the Netherland (ECN)

PYRENA is a novel integrated system that features ONE reactor in which fast pyrolysis, gasification and combustion can be combined. PYRENA is suitable for (catalytic) fast pyrolysis and offers several benefits over existing state-of-the art fast pyrolysis reactors such as continuous catalyst recycling, combination of in-situ and ex-situ catalysis, combination of pyrolysis, gasification and combustion in one heat-integrated system. Especially the flexibility regarding the use of catalysts is an attractive option, because it offers a route (via in-situ and ex-situ catalysis) to a better bio-oil that probably requires less downstream upgrading when compared to analogous pyrolysis systems. Better bio-oil means a fast pyrolysis bio-oil that is more stable, less acidic and relatively low in oxygen. These characteristics enable the use of this bio-oil as a good precursor for a drop-in co-feed in existing oil-refineries for the production of transportation fuels. Alternatively, the enhanced fast pyrolysis oil quality opens up several possibilities for extracting value added chemicals such as sugars, furanics and phenols.

PYRENA is deployed in a current Dutch national CatchBio – Catalytic Pyrolysis project to produce bio-oil samples for the project consortium and to tests catalysts. The CatchBio project deals with the application of catalysis in dedicated pyrolysis reactors aiming at improving the quality of the produced oil. Existing knowledge on catalytic pyrolysis serves as a starting point to study and optimize the catalyst in continuous reactors at a scale of 0.1 – 5 kg biomass per hour. Finding the best reactor configuration for catalytic pyrolysis is an integral part of the work. One of the target applications of the oil is a refinery co-feed. The main result of the project will be the identification of the best catalyst– technologies combinations for which a techno-economical evaluation and environmental assessment will be performed. PYRENA is a promising candidate.

PYRENA has successfully participated in a recent international round robin test on fast pyrolysis [1]. PYRENA bio-oil from poplar and wheat straw was nicely comparable with other state-of-the art pyrolysis reactors. The PYRENA set-up has been modelled in a coarse techno-economic evaluation, aimed at a scale of 300 kt/yr of dry biomass intake. Currently, PYRENA is used to study the effect of the environmentally benign mineral olivine as a mild, cheap and easily regenerable cracking catalyst on the fast pyrolysis of softwood.

This research has been performed within the framework of the CatchBio program. The authors gratefully acknowledge the support of the Smart Mix Program of the Netherlands Ministry of Economic Affairs and the Netherlands Ministry of Education, Culture and Science.

[1] D.C. Elliott, D. Meier, A. Oasmaa, B. van de Beld, A.V. Bridgwater, M. Marklund, K. Toven, “Results of the IEA Round Robin on fast pyrolysis bio-oil production”, Poster presentation on tcbiomass2015 conference, November 2015, Chicago, USA.

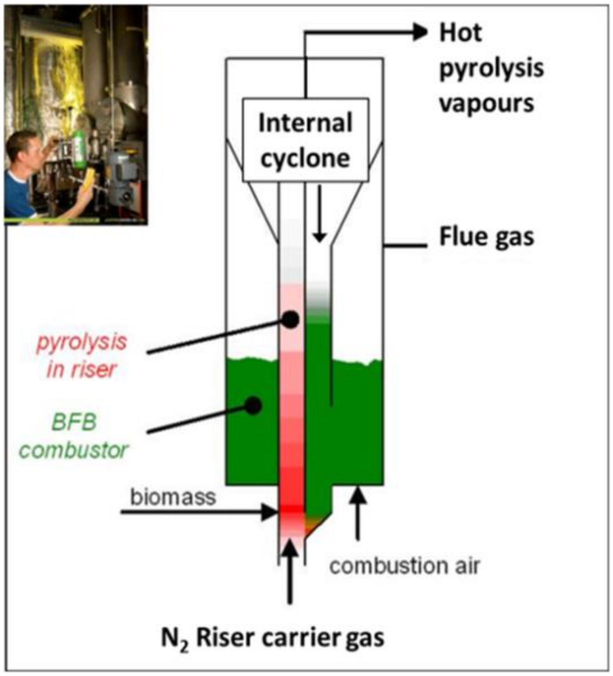

Figure 1: PYRENA consists of a bubbling fluidised bed reactor vessel, in the center of which an internal riser reactor is located for the pyrolysis, see the figure. Biomass is fed to the riser reactor at a maximum feed rate of 5 kg/hr and pyrolyzed at 400 – 600°C. The required heat is provided by hot sand or catalyst that circulates from the surrounding bubbling bed into the riser. The sand is heated via the combustion or gasification of the pyrolysis char that is transported from the riser reactor via an internal cyclone. The hot pyrolysis vapours are quickly quenched and recovered via direct or fractionated condensation. The PYRENA system is roughly comparable to a circulating fluidised bed (CFB) system. A major difference is the integration; most state-of-the-art CFB reactors use an external reactor for combustion of the char and heating of the sand.

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* presented at the 21th International Symposium on Analytical and Applied Pyrolysis in Nancy, France, 9 – 12 May 2016.

Converting Biomass Pyrolysis Vapors to Fungible Hydrocarbons using a Coupled Pyrolyzer Davison Circulating Riser System

Kim Magrini-Bair
National Renewable Energy
Laboratory (NREL)

In Department of Energy, Bioenergy Technologies Office (BETO) funded work, NREL partnered with W.R. Grace, a leading supplier of petroleum refining technologies, and Zeton Inc., a recognized world leader in pilot plant fabrication, to build a unique system that can produce fuel intermediates from biomass-derived liquids and vapors and can be utilized to demonstrate the co-processing of biomass-derived streams with petroleum. Identifying this critical pilot scale capability to develop refinery compatible biomass-derived fuel intermediates began late in 2012. This complex project included a year of design work to couple biomass vapor production with a riser reactor for catalytic upgrading to liquid fuel intermediates.

Concurrently, facilities upgrades at NREL began to house the pilot plant in a state of the art high bay laboratory. The plant shown below (Figure 1) was delivered to NREL in April 2014 and was fully operational in September 2014 after extensive training and commissioning were completed. In early 2015 co-processing verification experiments with vapor and petroleum showed that biomass components were integrated into the liquid gasoline product. In September 2015, whole vapors were upgraded to an aromatic and paraffinic product with low oxygen content.

Current work is evaluating the impact of catalyst type on product composition with catalysts tailored for pyrolysis vapors by Johnson Matthey, WR Grace, and NREL. These “bio-fuels” can then be finished in petroleum refineries to conventional fuels. The ability to leverage the existing U. S. petroleum refining infrastructure to produce “drop-in” biofuels (substitutes for conventional

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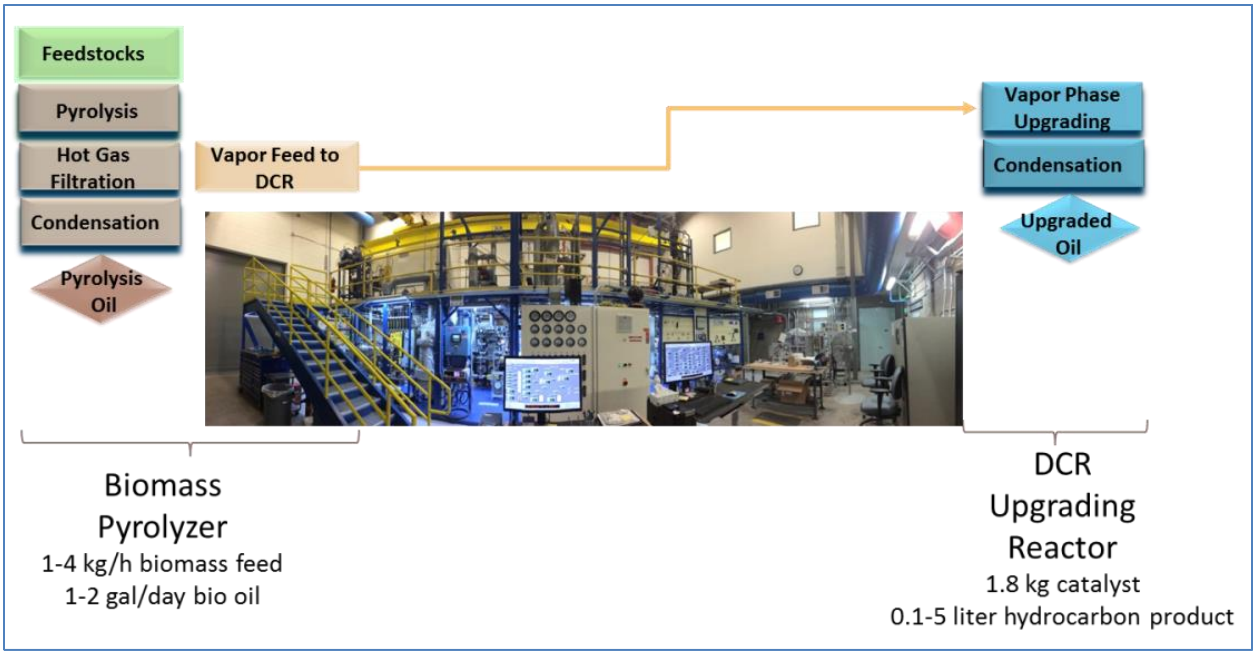

Figure 1: NREL’s coupled pyrolyzer/DCR system for upgrading biomass-derived pyrolysis liquids and vapors to fuels and chemicals. .

Converting Biomass Pyrolysis Vapors to Fungible Hydrocarbons using a Coupled Pyrolyzer Davison Circulating Riser System...continued

gasoline, diesel, and jet fuel) may provide significant reduction in the cost of biofuels. The goal of this project is to provide “biogenic refinery feedstocks” from biomass pyrolysis vapors using refinery accepted processing.

This new capability is based on fluid catalytic cracking (FCC) technology, the center of petroleum refining, as it can accept and reprocess a wide variety of feedstocks to gasoline. W.R. Grace is a leader in FCC development and its Davison Circulating Riser Reactor (DCR) is the pilot plant of choice for use by refineries worldwide to assess FCC operations and catalyst activity. The NREL system (shown in Figure 1) consists of a custom biomass pyrolyzer that produces vapors, which are upgraded with catalysts to fuel intermediates in the DCR. New catalysts designed for biomass vapors are developed and provided by our partner, Johnson Matthey. New feedstocks, developed by Idaho National Lab, are tested in the pyrolyzer. High quality data is generated using a variety of conditions in both systems on vapor and fuel product composition with rugged online instruments. This data is refinery compatible as are the liquid products. Processing upgraded liquids in the 110 domestic FCC units currently operating in the United States could produce greater than 8 billion gallons of bio derived fuels, directly addressing the renewable fuel standards set by the Energy Independence and Security Act of 2007.

In June 2016, the current round of upgrading catalyst testing was completed with the best performing material moving forward to pilot sale production for testing in NREL’s plant that operates at half a ton of day of biomass conversion and is 40 times larger than the DCR plant. 2-dimensional GCGCxTOFS spectra of liquid product from catalytic upgrading of E-Cat, VGO and nitrogen (vapor surrogate) compared with E-Cat, VGO and hardwood-derived pyrolysis vapor shows enhanced 1- and 2- ring aromatics and paraffins and isoparaffins formation from co-processing with pyrolysis vapors (Figure 2). The plant will next be upgraded for fractional product condensation that will be used to isolate specific products for chemicals production from vapors. The DCR system is available for industry to test their process concepts.

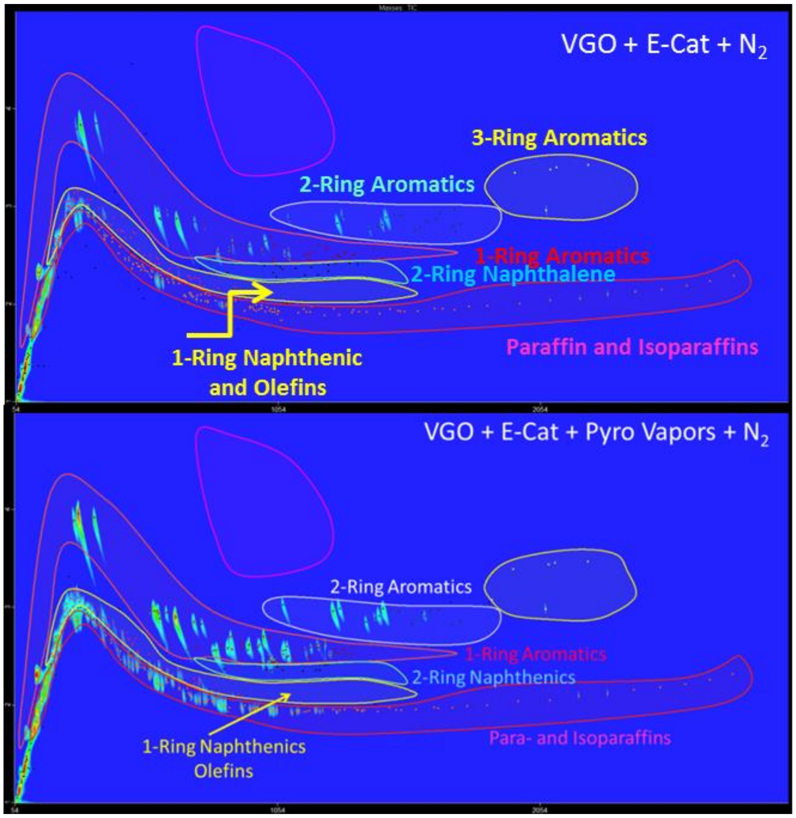

Figure 2: 2D GCGCxTOFS spectrum of liquid product from catalytic upgrading with E-Cat, VGO and nitrogen (top panel) compared with E-Cat, VGO, pyrolysis vapors and nitrogen (bottom panel). .

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NATIONAL RENEWABLE ENERGY LABORATORY

Hydrothermal Liquefaction of Wastewater Treatment Plant Solids

Justin M. Billing
Pacific Northwest National Laboratory (PNNL)

Feedstock cost is the greatest barrier to the commercial production of biofuels. The merits of any thermochemical or biological conversion process are constrained by their applicability to the lowest cost feedstocks. At PNNL, a recent resource assessment of wet waste feedstocks led to the identification of waste water treatment plant (WWTP) solids as a cost-negative source of biomass. WWTP solids disposal can account for up to half of WWTP operating costs [1], depending on the disposal method. Landfilling and land application may present environmental issues [2, 3] and be subjected to further regulation. The high moisture content is well-suited for hydrothermal liquefaction (HTL), avoiding the costs and substantial energy consumption associated with incineration or other dry reforming processes (e.g., pyrolysis). Testing at PNNL demonstrated that the yield and quality of biocrude and upgraded biocrude from WWTP solids may be comparable to that obtained from algae feedstocks but the feedstock is available at zero or negative cost as compared to over $1000 per dry ton for algae [4].

A collaborative project was initiated and directed by the Water Environment & Reuse Foundation (WERF) and included feedstock identification, dewatering, shipping to PNNL, conversion to biocrude by HTL, and catalytic hydrothermal gasification of the aqueous byproduct. Additional testing at PNNL included biocrude upgrading by catalytic hydrotreatment, characterization of the hydrotreated (HT) product, and a preliminary techno-economic analysis (TEA) based on empirical results. This short article will cover HTL conversion and biocrude upgrading. The WERF project report with complete HTL results is now available through the WERF website [5]. The preliminary TEA is available as a PNNL report [1].

HTL Conversion

Three types of WWTP solids were tested in bench scale HTL experiments: primary sludge, secondary sludge, and digested solids (biosolids). These streams were chosen to represent solids available within typical WWTPs. Sludge samples were dewatered and shipped to PNNL by Metro Vancouver from the Annacis Island WWTP. Properties of the sludge samples are given in Table 1. Primary sludge and digested solids were easily dewatered at Metro Vancouver up to or beyond the nominal processing target of 20 wt% solids. Water was added to the as-received solids for these streams to form a readily pumpable slurry. The resulting solids concentration was conservative and higher concentrations may be possible. The secondary sludge was challenging to dewater and a final concentration of 9.7 wt% was achieved.

Each sludge sample was tested at baseline HTL conditions (350°C, 2900 psig) in a continuous-flow hybrid reactor configuration including a CSTR and a plug flow reactor in series. A description of the PNNL bench scale system can be found in the WERF report or in other publications [6]. Following sludge liquefaction in the plug flow reactor, the stream was separated into products with the inline removal of precipitated solids, cooling, depressurization, and gravity separation of the biocrude from the aqueous phase. The reactor runs were routine and were characterized by stable operations, with the exception of the latter part of the

(Continued on page 18)

Table 1. Properties of WWTP Sludge Feedstocks

	Primary Sludge	Secondary Sludge	Digested Solids
Solids [wt%]	11.9	9.7	16.0
Ash [wt%]	7.5	16.2	28.1
Solids [wt% daf]	11.0	8.1	11.5
Density [g/cm^3]	1.04	1.00	1.05
pH	5.1	6.0	7.7
C [wt%]	47.8	43.6	38.7
H [wt%]	6.50	6.55	5.68
O [wt%]	33.6	29.0	27.9
N [wt%]	3.64	7.90	4.48
S [wt%]	0.48	0.72	1.63
HHV [MJ/kg]	20.7	19.6	16.8

Note: CHN, O, S, HHV, and ash values on a dry basis. daf: dry, ash-free

Hydrothermal Liquefaction of Wastewater Solids…continued

secondary sludge run where there was a suspected loss of mixing leading to solids accumulation in the CSTR.

For primary sludge, the biocrude yields on both carbon and mass bases were similar to what has been observed for algae feedstocks, as shown by the comparison to Tetraselmis sp. marine algae in Figure 1. The yield is calculated against the total mass or carbon mass in the dry, ash-free (daf) biomass feedstock. The mass and carbon yields to biocrude from digested solids were reasonable (34 and 49 %, respectively), while yields were somewhat lower for secondary sludge (25 and 39 %, respectively). This may have been due to the low starting solids concentration that led to emulsions in the product stream, potentially compounded by operational difficulties (loss of mixing). The secondary sludge stream would benefit from blending with the primary sludge stream, both for dewatering and hydrothermal processing.

In addition to the yield, the biocrude quality from primary sludge was comparable to Tetraselmis sp. biocrude. Table 1 contains analytical data for biocrude and upgraded HT product for each feedstock. Nitrogen and sulfur content was lower in the sludge biocrude, but the oxygen content was higher. The biocrudes have similar energy content (HHV) and density, though the viscosity was slightly higher for the sludge biocrude. Insoluble solids content was higher than the algae biocrude but still acceptably low for gravity-separated biocrude. Elemental compositions and ratios are calculated on a dry basis, while density, viscosity, and filterable solids are measured on the whole biocrude, with moisture included. Across nearly a dozen categories, the primary sludge biocrude was of the same quality as the algae-derived biocrude.

Table 2. Comparison of chemical and physical properties of HTL biocrude and upgraded HT product from primary sludge and *Tetraselmis* sp. marine algae

Analysis	**Primary Sludge**		**Tetraselmis**	
	Biocrude	HT Product	Biocrude	HT Product
Carbon [wt%]	76.5	84.0	79.4	87.0
Hydrogen [wt%]	10.1	14.5	10.1	14.9
H:C atomic ratio	1.6	2.0	1.5	2.1
HHV [MJ/kg]	37.8		39.2	
Nitrogen [wt%]	4.3	0.05	4.8	<0.05
Oxygen [wt%]	8.4*	1.2	3.8	0.9
Sulfur [ppm]	6300	23	14,000	15
Moisture [wt%]	13.0	<0.5	6.6	<0.5
Density [g/cm^3]	1.00‡	0.79†	0.99‡	0.78†
Viscosity [cSt]	571‡	2.5†	245‡	1.4†
TAN [mg $_{KOH}$/g]	65	< 0.01	54	<0.01

*Oxygen by difference †At 20°C ‡At 40°C

Biocrude Upgrading

The biocrude from primary sludge and digested sludge was catalytically hydrotreated (HT) to remove oxygen, nitrogen, and sulfur in a bench scale trickle flow reactor. The single-step reaction was carried out at 400 °C and 1540 psig in the presence of hydrogen using a sulfided catalyst (Co-Mo/Al2O3, Alfa Aesar). The HT product was substantially deoxygenated, denitrogenated, and desulfurized and had a density of 0.79 g/cm3 (see Table 2). The H:C molar ratio

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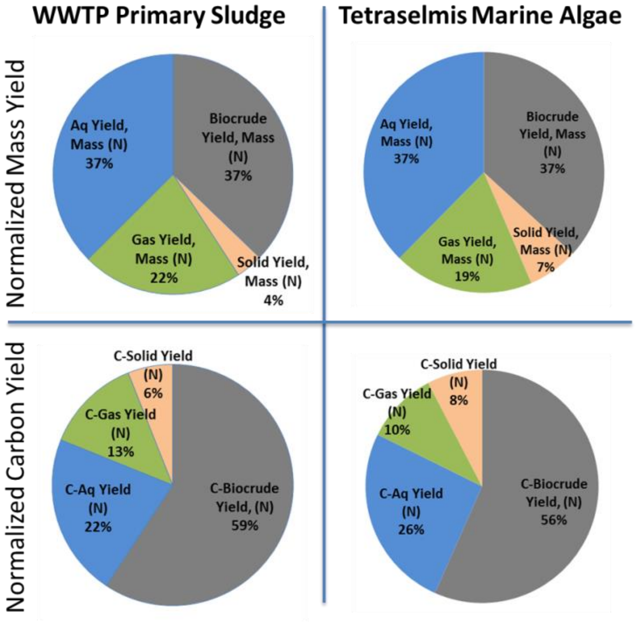

Figure 1. Normalized mass and carbon yields from WWTP primary sludge and Tetraselmis marine algae. Yields are computed on a dry, ash-free (daf) basis and normalized by mass balance.

Hydrothermal Liquefaction of Wastewater Solids…continued

was 2.0, indicating a hydrocarbon product with a limited number of heteroatoms and double bonds, suitable for fractionation into fuel-range products or integration with existing refinery streams. Like the HTL biocrude, the sludge-derived HT product had similar physical and chemical characteristics to algae-derived HT products, including boiling point range as determined by simulated distillation. Additional characterization data will be the subject of future reporting.

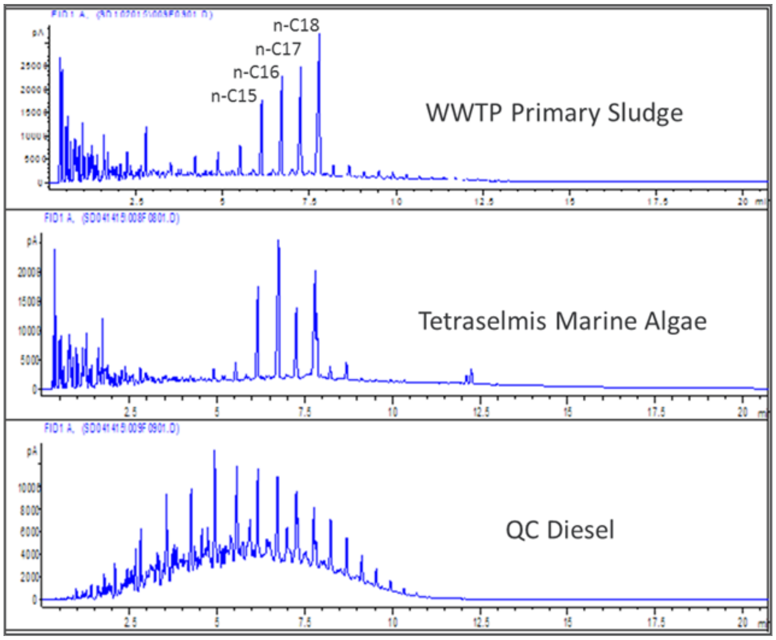

Figure 2. GC-FID chromatograms for hydrotreated biocrude from WWTP primary sludge and Tetraselmis marine algae compared with a diesel reference sample. The primary sludge shares the signature hydrocarbon series from n-C15 to n-C18, among other features.

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Task 34 Progress and Plans

Alan Zacher
Task 34 Lead

A host of changes followed the IEA Bioenergy Task 34 into the 2016 to 2018 Triennium. The title of the new Task will be Direct Thermochemical Liquefaction, and is an opportunity to bring a comprehensive overview of the various technologies used to produce thermochemically liquefied biomass for fuels and chemicals.

The scope of activities will be expanded to include hydrothermal liquefaction and the upgrading of bio-oil and biocrude to hydrocarbon liquid fuels alongside fast pyrolysis. The participants committed to the Task for this triennium are Canada, Finland, Germany, the Netherlands, New Zealand, and Sweden, with leadership provided by the USA.

The objective of Task 34 is to facilitate commercialization of liquid fuels from biomass through direct thermochemical liquefaction, particularly fast pyrolysis and solvo-thermal processing. Our goal is contributing to the resolution of critical technical areas and disseminating relevant information particularly to industry and policy makers. Our scope is to monitor, review, and contribute to the resolution of challenges that will permit more successful and more rapid implementation of biomass liquefaction technology, including identification of opportunities to provide a substantial contribution to bioenergy.

The following are the Priority Topics identified for the Triennium by the Task:

- Provide support for commercialization through standards development;
- Validate applicable analytical methods for product evaluation;
- Facilitate information exchange with stakeholders;
- Support technoeconomic assessment of liquefaction technologies.

Progress

During 2016, Task members have been working on:

- The Task has a new webpage: task34.ieabioenergy.com/ that will be developing over 2016.
- Draft manuscript of the IEA Round Robin on Fast Pyrolysis Bio-oil Production is near completion. Initial results were presented at TCBiomass in 2015, and this manuscript represents the complete results. It summarizes the work of fifteen laboratories in six different countries converting three different biomass samples into bio-oils via fast pyrolysis. Samples of the bio-oil were analyzed at a central laboratory to validate the pyrolysis community understanding of production of fast pyrolysis bio-oil by providing a common feedstock for bio-oil preparation. The results showed that the bio-oil products were fairly consistent when produced in a fluidized bed fast pyrolysis system, while other reactor configurations produced products with somewhat different properties.
- Support to the CEN working group continues developing standards for bio-oil for use in Europe. Two of the Task members are members of the working group (see page 3).
- There are new updates to the web-based interactive database of pyrolysis demonstration plants which is now an active element on the Task website (see page 21).
- The first team meeting of Task 34 was to be held on June 13 in Luleå/Piteå, Sweden, but was postponed due to travel disruptions. This was unfortunate, as also postponed was a domestic workshop in thermochemical liquefaction and upgrading of biomass commissioned by the Bothnia Bioindustries Cluster (BOBIC) with a well-planned program including talks on Bioenergy commercialization and complementary research presentations, along with tours of Sweden's active bioenergy research and development.
- Planning has been completed for the 2nd task meeting of 2016, to be held in Rotorua, New Zealand on 7-8 November. This includes a comprehensive workshop

(Continued on page 21)

Pyrolysis Demoplants Database

The IEA Bioenergy Task 34 website - task34.ieabioenergy.com/ - has a new resource available: The Pyrolysis Demoplants Database.

The searchable tool contains a worldwide database of pyrolysis plants around the world capturing Technology type, Technology Readiness Level (TRL), and current Status. This data can be viewed as a list or in an interactive map from the webpage.

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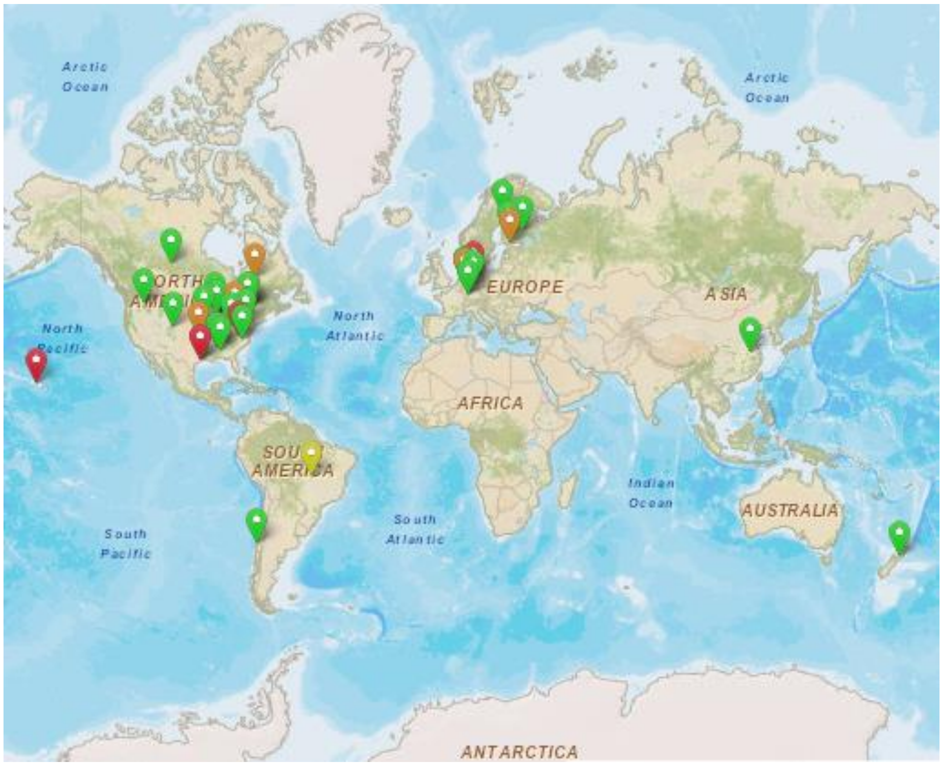
Work on the database is ongoing and if you are aware of any plants not currently listed, contact Alan Zacher at alan.zacher@pnnl.gov.

To access the database page, visit

task34.ieabioenergy.com/



Task 34 Progress and Plans...continued

(Continued from page 20)

hosted by IEA Bioenergy ExCo78 on Marine and Aviation Biofuels. The week will finish off with an ABRN science symposium in International Biofuels Developments in which Task members are giving talks and participating in panel discussions.

- Task 34 NTL of Germany, Nicolaus Dahmen led an exploratory tour of bioenergy companies and researchers in the USA to establish closer collaborations in the area of biomass thermochemical conversion for synthetic fuels and chemicals.
- An updated Pyrolysis brochure, such as published by the Task in 2007 is in discussion.
- An initial integrated framework for the various liquefaction technologies is being developed, which will likely result in an additional brochure covering a spectrum of liquefaction methods.

In this issue of the newsletter there are an excellent mix of articles on both pyrolysis and solvo-liquefaction.

Please examine the Country Reports located elsewhere on the Task 34 website. These are short introductory articles and slide sets prepared by the national team leaders from each of the participating countries summarizing the particular biomass pyrolysis efforts in their countries. In addition, the latest Task meeting report summarizes the developments within each of the Priority Topics of the Task.

Documentation will be found on both the task34.ieabioenergy.com/ as well as our original site during the transition. We welcome your thoughts on resources to include serving the direct thermochemical liquefaction community.

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For Further Information

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Pyrolysis Demoplant Database
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If you would like to contribute an
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Task 34

Direct Thermochemical Liquefaction

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