IEA Bioenergy

Task 34 - Pyrolysis

Welcome to Task 34

By Doug Elliott, Task 34 Leader

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The IEA Bioenergy Task 34 for Pyrolysis has completed the current Triennium, which ran from 2013 to 2015. Participants in the Task were Germany, the Netherlands, Sweden, Finland, the UK, and Norway with leadership provided by the US. The national team leaders from the participating countries are listed on page 2 of this newsletter.

With the end of the Triennium the Task will undergo a significant transition, both in its scope and also as two long-term participants undergo transitions in their professional lives. I will pass the leadership of the Task to Alan Zacher of Pacific Northwest National Laboratory beginning January 1, 2016, as I retire effective January 31, 2016. The national team leadership role in Germany will change to Nicolaus Dahmen of Karlsruhe Institute of Technology as Dietrich Meier from Thünen will be retiring from the Institute early in 2016.

IEA Bioenergy Task 34 members and colleagues at meetings that took place during this Triennium.

Aims & Objectives

The overall objective of Task 34 during the past Triennium was to improve the rate of implementation and success of fast pyrolysis for fuels and chemicals by contributing to the resolution of critical technical areas and disseminating relevant information, particularly to industry and policy makers. The following were the Priority Topics identified for the Triennium by the Task:

- Review of bio-oil applications;
- Bio-oil standardisation;
- Round Robin for analytical method development;
- Technoeconomic assessment of thermochemical liquefaction technologies.

Progress

During the current Triennium, the Task members have been busy with the identified activities.

- The Applications for bio-oil were reviewed and new information was incorporated into a new IEA Bioenergy document on pyrolysis, to be released early next year.
- The Round Robin tests were completed at the participating laboratories and analysis of the

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Welcome ...continued

samples was undertaken at the Thünen Institute in Hamburg, Germany. Three feedstocks were prepared and distributed and 16 laboratories prepared fast pyrolysis bio-oils for analysis and comparison. The results of the Round Robin have been prepared into a draft manuscript, which is being reviewed for publication.

- Support to the CEN working group, which is developing standards for bio-oil for use in Europe, has been ongoing, with two of the Task members actually serving as members of the working group, while all Task members serve as technical consultants to the standards development effort.
- In recent collaborations with other IEA Bioenergy Tasks the life cycle analysis produced by two of the Members was reviewed in Task 38 and other participants in Task 34 have provided review of the pyrolysis-based biorefinery fact sheet being developed as part of the Task 42 work.
- Finally, a new web-based interactive database of pyrolysis demonstration plants was developed and is now an active element on the Task website (see page 33).

The progress in the field of biomass pyrolysis has been dramatic over the past three years as demonstrated by the start-up of commercial fast pyrolysis systems in both Finland and the Netherlands, while in North America commercial multi-year contracts were signed and bio-oil delivered from Canada to three different customers in the US to replace fossil fuels for heat production.

In this issue of the newsletter there are several articles from the participants describing the latest developments in fast pyrolysis, including updates from Finland, Sweden, Germany, the UK and the US, alongside articles from two non-member countries: Thailand and China. There is an article from the Task Representative for New Zealand, which will be joining the Task in the new year, and an insight into Task 34's plans for the new Triennium. There is also an updated calendar of events of interest to the biomass pyrolysis community.

Please also be aware of 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 summarising the particular biomass pyrolysis efforts in their countries. In addition, the latest Task meeting report summarises the developments within each of the Priority Topics of the Task.

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Scion joins IEA Bioenergy Task 34 as New Zealand representative

Ferran de Miguel Mercader (above) and Kirk Torr (below) of Scion discuss representing New Zealand in Task 34 as they become a Task Member in the new Triennium

Figure 1: Scion's pyrolysis plant.

Scion is joining Task 34 (Direct Thermochemical Liquefaction) in the new Triennium as New Zealand representative. Scion is a government-owned research institute that concentrates on improving the international competitiveness of the New Zealand forest industry and building a stronger biobased economy. This includes a range of bioenergy and biorefinery research and development activities across the whole value chain, from resource establishment through to product development.

Scion has been working in the bioenergy and biofuels area for several years, with focus on developing technology to use biochemical routes for converting softwoods to biofuels and chemicals, particularly looking at pretreatment and enzymatic saccharification. The sugars produced from softwood can be fermented to biofuels, biochemicals and bioplastics.

Recently, a programme investigating thermochemical technologies to transform biomass into fuels and chemicals was started at Scion. The programme's goals are supporting industry to accelerate the implementation of "drop-in" biofuels in New Zealand, and to take advantage of co-production of chemicals and materials. Since the start of the thermochemical technologies programme, four capability areas have been developed: solid wood upgrading (e.g. torrefaction); bio-oil characterisation; bio-oil production; and thermodynamic and techno-economic analysis. A fifth area on bio-oil upgrading is under development.

Bio-oil production at Scion

A micro-reactor system and a pyrolysis plant have been established at Scion for the production of bio-oils.

The micro-reactor system is very

(Continued on page 4)

Scion joins IEA Bioenergy Task 34 *...continued*

Figure 2: Scion's micro-reactor system.

versatile and capable of carrying out experiments at high temperatures (up to 400°C) and high pressures. The system can be used for fast screening (several experiments per day) of different processes, conditions and feedstocks. Small reactors (typically 15mL) can be loaded with solid and/or liquid samples. Gases (e.g. nitrogen or oxygen) can also be added to carry out different types of reactions (e.g. hydrothermal liquefaction or wet oxidation). Using a robotic arm controlled from a separate room, the loaded reactor is introduced into a hot fluidised sand bed. Very high heating rates are achieved (as fast as 300°C/min), significantly reducing the effect of heating time on the experimental results. The features of small size, automation and separation between operator and reactor all contribute to a high level of operator safety.

Scion's fast pyrolysis plant was designed by the University of Twente and built by Head Consultancy BV. It is capable of converting 1kg/h of wood into pyrolysis oil using fluidised bed technology. The plant can carry out catalytic or non-catalytic pyrolysis experiments.

The pyrolysis plant is equipped with two types of condenser systems: dual spray columns and an electrostatic precipitator (ESP). The spray columns use paraffin oil as cooling media and can be operated at different temperatures, allowing two fractions of differing volatility to be obtained. The ESP is specially designed to run catalytic pyrolysis experiments, as otherwise the paraffin oil used in the spray columns would dissolve part of the product. Currently, the plant is being used to evaluate the effect of biomass pretreatment on the oil properties and the oils' further upgradability.

Bio-oil characterisation

A complete suite of analytical methods has been established to characterise the experimental bio-oils produced, including:

- Elemental analysis (C,H,N and O);
- Water content (Karl Fischer);
- Total acid number (Organic titration);
- Heating value (Bomb calorimeter);
- Inorganic content (IC-MS);
- Molecular weight distribution (GPC);
- Solvent fractionation (developed by VTT);
- Functional groups (^{1}H, ^{13}C & ^{31}P NMR).

Pyrolysis gas chromatograph-mass spectrometry (Py-GC-MS) for wood has also been developed and is being used to compare catalytic and non-catalytic pyrolysis of *Pinus radiata* wood.

Scion's thermochemical technologies team is enthusiastic about joining IEA Bioenergy Task 34 as a way to collaborate with international partners and contribute to the development of direct thermochemical liquefaction technologies for deployment into the market.

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Controlling the phase stability of biomass fast pyrolysis bio-oils

Anja Oasmaa summarises the recent paper published by herself and her colleagues at VTT, Finland

Fast pyrolysis bio-oil (FPBO) is a promising alternative to fossil fuels and is currently entering the heating oil market.[1] These bio-oils are typically single-phase liquids and work on norms and standards for their use is ongoing.[2] However, in some cases phase-separation can take place. In a recent paper[3] we present how to control the phase stability of biomass fast pyrolysis bio-oils and a summary is presented here.

High extractive content of the feedstock

Phase separation as a formation of a waxy hydrophobic upper phase (Figure 1A) can be observed in bio-oils from extractive-rich feedstocks, like forest residues. The top phase is very rich in extractive compounds and contains solids (char), but also small quantities of other bio-oil components depending on the condensation conditions (i.e. temperature, time). Solid char particles adhere to the sticky extractives and rise up to the surface of the bio-oil. The stratification to top and bottom phases is slow (about 24 hours at 35°C) and can be avoided by constant mixing.

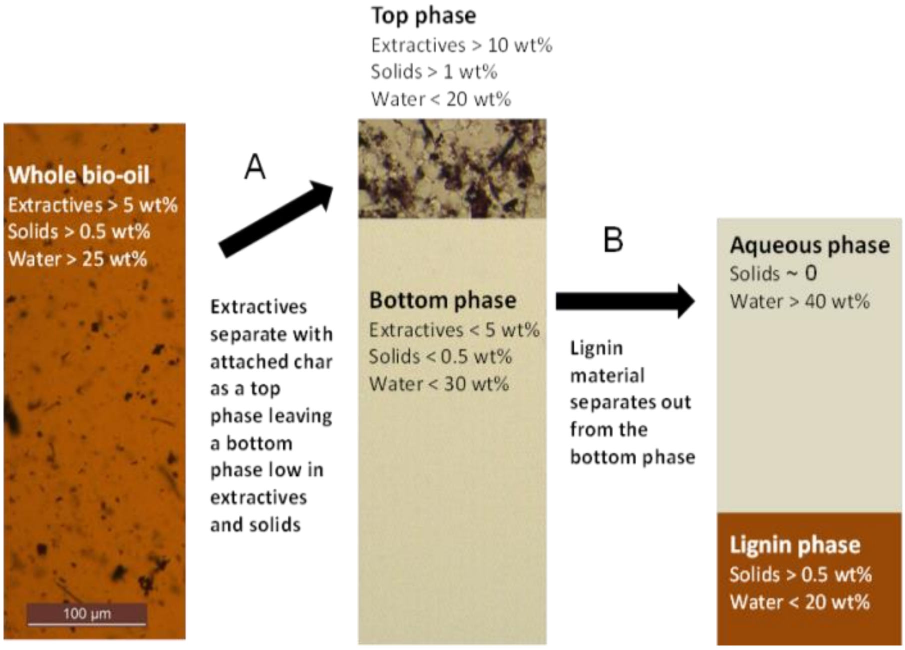

Figure 1: Phase separation of forest residue fast pyrolysis bio-oil.

High water or ash content of feedstock

Fast pyrolysis bio-oils can dissolve around 30wt% water, above which a phase-separation to an aqueous upper phase and viscous lignin-phase takes place (Figure 1B). This can be due to feedstock that is too moist or high in ash content. High ash content of agrobiomass leads to bio-oils with higher water content and lower oil yield due to the catalytic effect of alkali metals in the ash. Figure 2 shows the effect of feedstock ash content on

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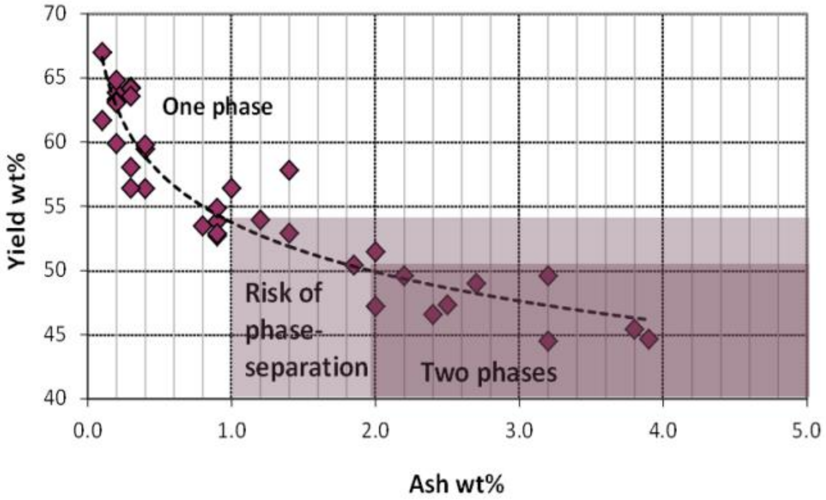

Figure 2: Effect of feedstock ash on phase separation tendency of fresh bio-oil. The curve is based on numerous experimental data points with wood and agrobiomass obtained by VTT's 20 kg/h pyrolyser.

Controlling the phase stability ...continued

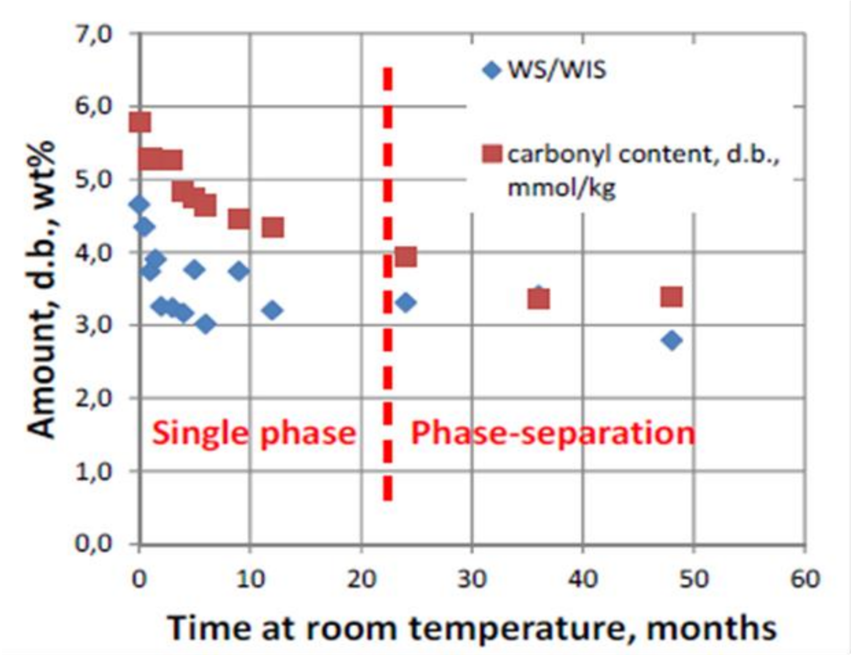

Figure 3: Phase-stability of a pine bio-oil (1) at room temperature. WS =Water-solubles, WIS= Water-insolubles, d.b. = dry basis.

the yield of organics and the phase separation tendency of fast pyrolysis bio-oils. This type of phase-separation can be controlled by specifying the maximum moisture and ash contents of feedstock.

Ageing

All fast pyrolysis bio-oils phase-separate at some point due to ageing[4] reactions causing, for example, an increase in the molecular weight and water-insoluble fraction (WIS), and reactions of carbonyl compounds leading to a decrease in carbonyl content. Figure 3 shows change in carbonyl content and the water-solubles/water-insolubles (WS/WIS) ratio of the pine pyrolysis bio-oil (1) by time and phase separation zone. The phase-separation due to ageing can be controlled by specifying bio-oil storage and handling temperatures and/or the addition of co-solvents, like alcohols.

Phase diagram for describing the phase stability of FPBO

To better understand the phase stability of fast pyrolysis bio-oils, a phase diagram was drawn based on the fact that the bio-oil is formed by three pseudo-components. The chemical composition of the bio-oil was described as a blend of a very hydrophilic fraction (water, anhydrosugars, polyols and other compounds with many hydroxyl groups), a very hydrophobic fraction (lignin degradation products, wood extractives) and a fraction that acts as a co-solvent (C1 – C6 oxygenated molecules that include aliphatic and aromatic acids, aldehydes, ketones, alcohols and phenols). Figure 4 shows the correlation between the chemical composition and the homogeneity of fresh and aged bio-oils in a ternary phase diagram. Blue circles and red squares represent one or two phase bio-oils, respectively. Their positions in the diagram show their chemical composition. Small arrows attached to the axes point the directions how to read the diagram.

The composition of normal fresh bio-oils is typically 55 : 20 : 25 (wt. ratio of water and sugars : WIS : co-solvents). After one year of ageing the composition changed to approximately 55 : 25 : 20, but the bio-oil remained homogeneous. After four to eight years of ageing the WIS content increased, the solvent content decreased to as low as 55 : 40 : 5 and the bio-oil separated to two phases. The change in chemical composition during ageing is highlighted with the blue dashed arrow in the diagram. Aged bio-oils appear in the bottom of the diagram because the amount of WIS content increases and the amount of C1 – C6 oxygenated compounds decreases during repolymerisation.

As the content of WIS increases, the ratio WIS/(sugars+H_2O) has to increase exponentially, mainly due to the need to replace part of the sugars+H_2O by a co-solvent capable of solubilising the WIS fraction. The proposed index is shown in equation 1:

$$Stability\ Index = \frac{10.66 \cdot WIS \cdot e^{(-0.0611 \cdot WIS)}}{Water+Sugars} \qquad (1)$$

Where:
WIS = Content of Water-Insoluble Fraction (wt.%)
Water+Sugars = Content of Water and Sugars (wt.%)

This index can be used to predict the expected phase stability of bio-oils if the contents of water, sugars and water-insoluble fractions are known. A stability index value close to one indicates that the oil is very close to phase equilibrium. Consequently, very small amounts of water formed during ageing can result in the formation of two phases. A very high stability index indicates that the oil is unlikely to form separate phases during storage. A stability index below one is indicative of oils that will form at least two phases upon storage.

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Controlling the phase stability *...continued*

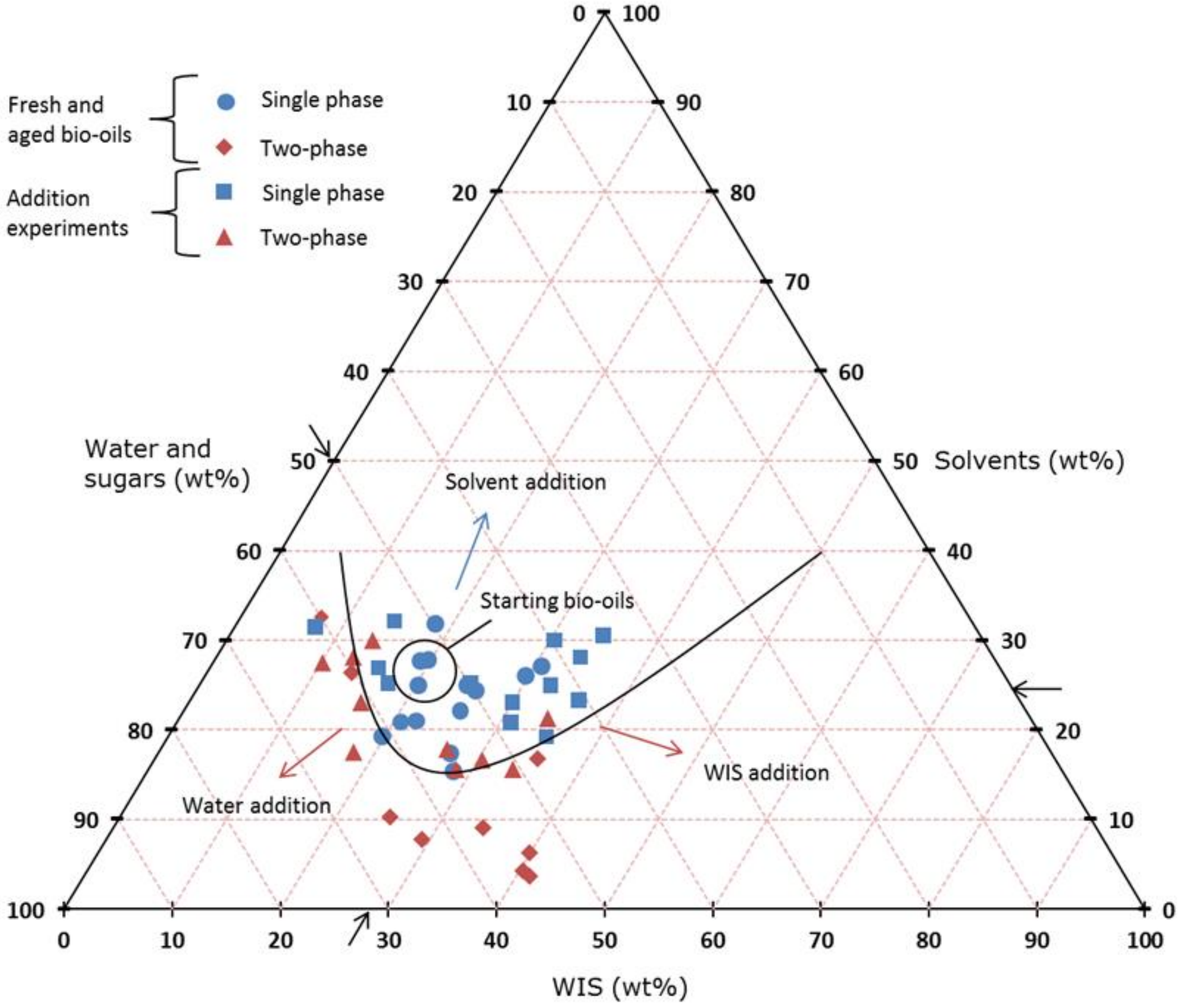

Figure 4: Ternary phase diagram of phase stability including additional experiments with water, water-insoluble (WIS) fraction and a model solvent mixture.

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Supercritical CO_2 extraction of value-added chemicals from pyrolysis liquids

Yongshun Feng of the University of Hamburg (left, top) and Dietrich Meier of Thünen Institute of Wood Research (left, bottom), Germany, discuss their new work on pyrolysis using supercritical CO_2

Bio-oil from fast pyrolysis has been drawing much attention owing to its potential to provide both energy and chemicals in new biorefinery concepts.[1] The application of bio-oil as a replacement for traditional chemicals is always a challenge due to its complex composition. Bio-oil is a liquid consisting of several hundred chemical components and most of the components are present in low concentration. Therefore separation or fractionation of bio-oil is necessary for further application. Our research group has studied an innovative extraction method with supercritical carbon dioxide ($scCO_2$). At supercritical state, CO_2 behaves like an organic solvent with a density comparable with gas. Solubility is therefore greatly improved and selective extraction can be tuned through variation of pressure and temperature, amongst other factors. The use of $scCO_2$ has several advantages: it is non-toxic, it becomes supercritical at 31°C (thereby creating no thermal stress for the sample), and the extract is solvent-free.

The extraction with $scCO_2$ has been successfully applied in commercial processes, such as decaffeination or extraction of essential oils from plant material. In our research, both slow pyrolysis liquid from beech wood and fast pyrolysis liquid from pine wood have been studied.[2] The slow pyrolysis liquid is a byproduct of a commercial charcoal company (ProFagus, Bodenfelde, Germany). If successful, our extraction method could make full use of this byproduct. Different pressures (100 -300bar), temperatures (60-80°C), and loadings were used to investigate extraction efficiency and selectivity.

Since the experimental setup does not allow for direct extraction of a liquid, solid carriers for the pyrolysis liquids were used developing different intermolecular forces between the liquid and the carrier. The extraction selectivity could potentially be changed by using different carriers. For the preliminary study, 100g silica gel (0.2–0.5mm) was used to adsorb 80g pyrolysis liquid. After mixing, they were transferred into a stainless steel basket, and inserted into the extractor. The extraction system (HDT Sigmar Mothes, Berlin, Germany) is shown in Figures 1 and 2. The extractor volume was 640mL (750long mm×33mm i.d.). CO_2 from a cylinder was delivered by a membrane pump first into the pre-heater to reach supercritical conditions, and then into the extractor. The CO_2 mass flow rate was set to 10g/min. The extraction pressure was controlled

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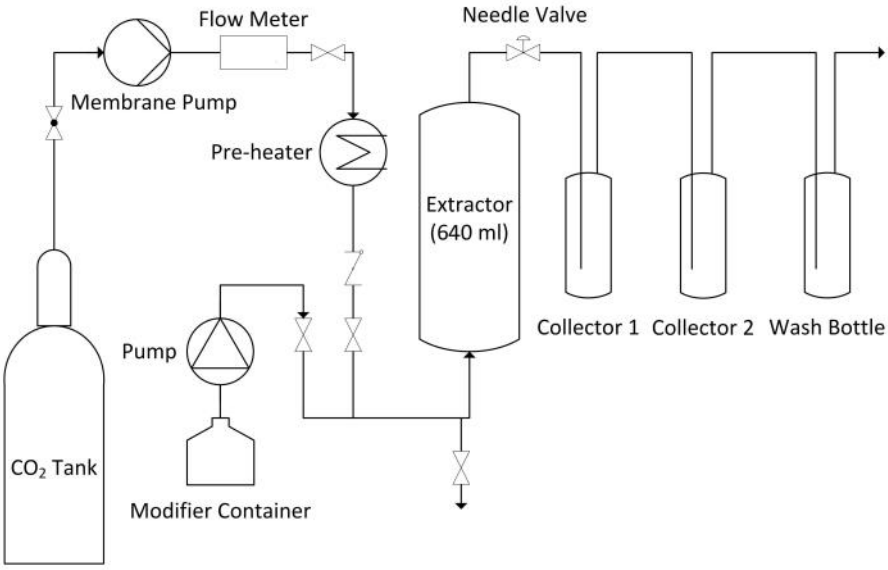

Figure 1: Schematic diagram of the supercritical CO_2 extraction system.

Supercritical CO_2 extraction *...continued*

Figure 2: The supercritical CO_2 extraction unit at Thünen Institute of Wood Research.

automatically by a back pressure regulator (Flowserve, Kämmer Ventile Type 81057-P). Extracts were collected every hour in glass collectors at ambient pressure.

After the scCO_2 extraction, residues were desorbed from the silica gel by Soxhlet extraction with acetone and subsequent removal of the solvent by rotary evaporation. Properties and chemical compositions were analysed for all starting pyrolysis liquids, extracts and residues by KF-water determination, elemental analysis, higher heating value calculation (HHV), gas chromatography coupled with mass spectrometry and parallel flame ionisation detection (GC-MS/FID), and gel permeation chromatography (GPC).

Transparent reddish extracts could be obtained after scCO_2 extraction due to their much lower viscosity, as shown in Figure 3. The extraction yield of water-free slow pyrolysis liquid was in the range of 40-52wt%, while for fast pyrolysis liquids the yield was only 7-12wt%. The extraction efficiency was largely dependent on the water content. The water content of the fast pyrolysis liquid upper phase decreased from 43wt% to 17wt% and for the lower phase it decreased from 19wt% to 12wt%. Changes of elemental composition and higher heating value (HHV) on a dry basis were insignificant.

Extracts are mostly composed of volatile low molecular weight components, while residues show much higher molecular weights compared to original pyrolysis liquids. As a consequence we used GC-MS/FID to obtain qualitative and quantitative data. Components in pyrolysis liquids and extracts were divided into ten groups, namely acids, esters, aldehydes, ketones, furans, pyrans, phenols, guaiacols, syringols and others, shown in Figure 4. Most of the value-added chemicals in the pyrolysis liquids could be enriched in the extracts such as acids, aldehydes, ketones, furans and aromatic compounds. However, the enrichment effect is not obvious for the esters, alcohols, aldehydes and pyrans. Pressure between 150 and 250 bar affects mainly the extraction yield rather than the composition, whereas the yield increased by approximately 10wt% for slow pyrolysis liquid and 4wt% for fast pyrolysis liquid.

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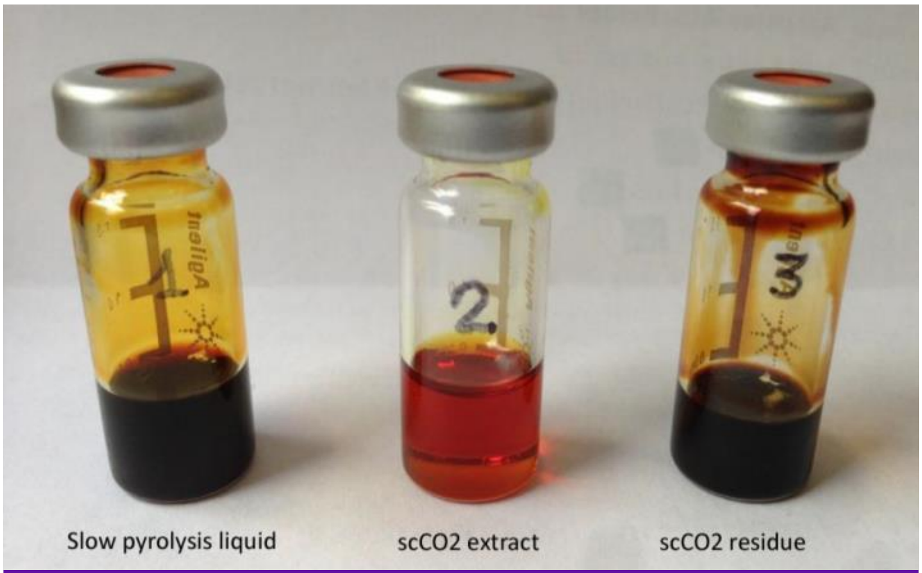
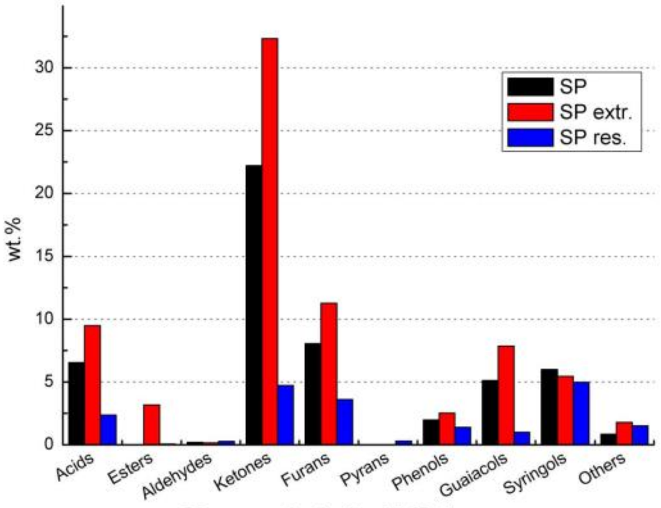
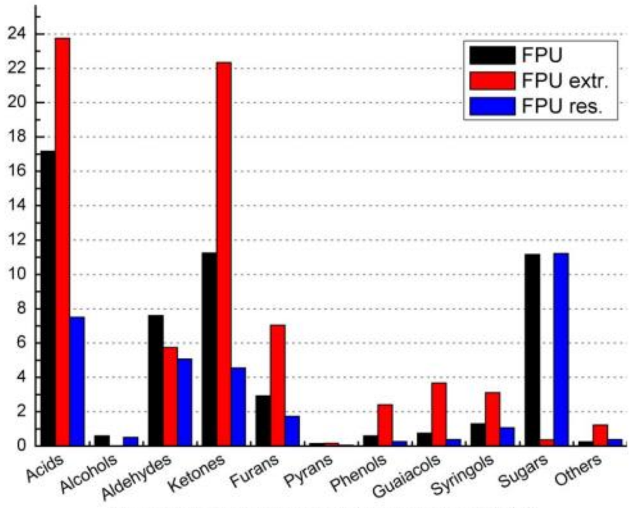


Figure 3: Slow pyrolysis liquid, scCO_2 extract and residue.

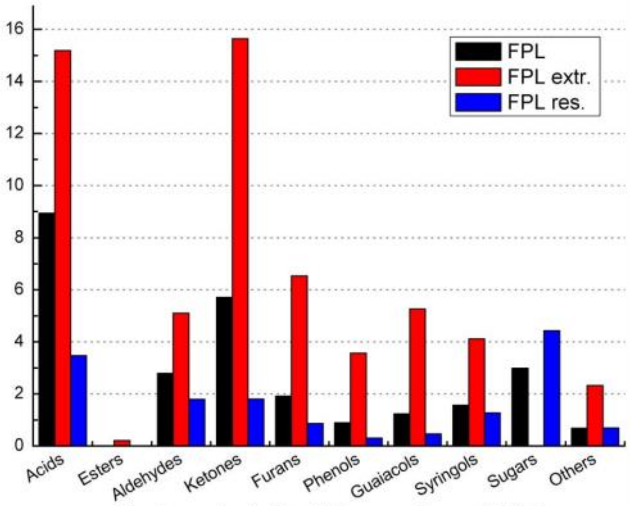
Supercritical CO_2 extraction *...continued*



Slow pyrolysis liquid (SP)



Fast pyrolysis liquid upper phase (FPU)



Fast pyrolysis liquid lower phase (FPL)

Figure 4: Distribution of chemical groups in pyrolysis liquids, $scCO_2$ extracts and residues.

Our study showed that $scCO_2$ extraction has a positive effect on the enrichment of value-added chemicals in pyrolysis liquids. Furthermore, the extracts exhibit improved physical properties (no phase separation, no solids, less viscosity, no pyrolytic lignin). On the other hand, selectivity still needs to be improved to acquire target chemicals in higher yield and purity. Future work will focus on different carriers, staged condensation at two pressure levels, and the use of commercial fast pyrolysis liquids.

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Hydrothermal liquefaction of biomass, revisited

As interested readers may remember, I originally drafted a report on this topic for the newsletter in 2010 (Issue 28). Here I have updated the original article with the latest developments. All of this is an introduction to liquefaction as an alternative biomass to the liquid fuels route, which will become part of Task 34 in the new Triennium, 2016-2018.

Doug Elliott of Pacific Northwest National Laboratory, USA, revisits his earlier article

Hydrothermal liquefaction (HTL), called high-pressure liquefaction in earlier years,[1] is an alternative process for conversion of biomass into liquid products. Some experts consider it to be pyrolysis in solvent phase. It is typically performed at about 350°C and 200atm pressure such that the water carrier for biomass slurry is maintained in a liquid phase, i.e. below super-critical conditions. In some applications catalysts and/or reducing gases have been added to the system with the expectation of producing higher yields of higher quality products. Slurry agents ("carriers") evaluated have included water, various hydrocarbon oils and recycled bio-oil. High-pressure pumping of biomass slurry has been a major limitation in the process development.

Process research in this field faded away in the 1990s except for the HydroThermal Upgrading (HTU) effort in the Netherlands,[2] but has new resurgence with other renewable fuels in light of the increased oil prices and climate change concerns. Research restarted at Pacific Northwest National Laboratory (PNNL) in 2007 with a project, "Hydrothermal Liquefaction of Agricultural and Biorefinery Residues" with partners Archer-Daniels-Midland Company and ConocoPhillips. Through bench-scale experimentation in a continuous-flow system this project investigated the bio-oil yield and

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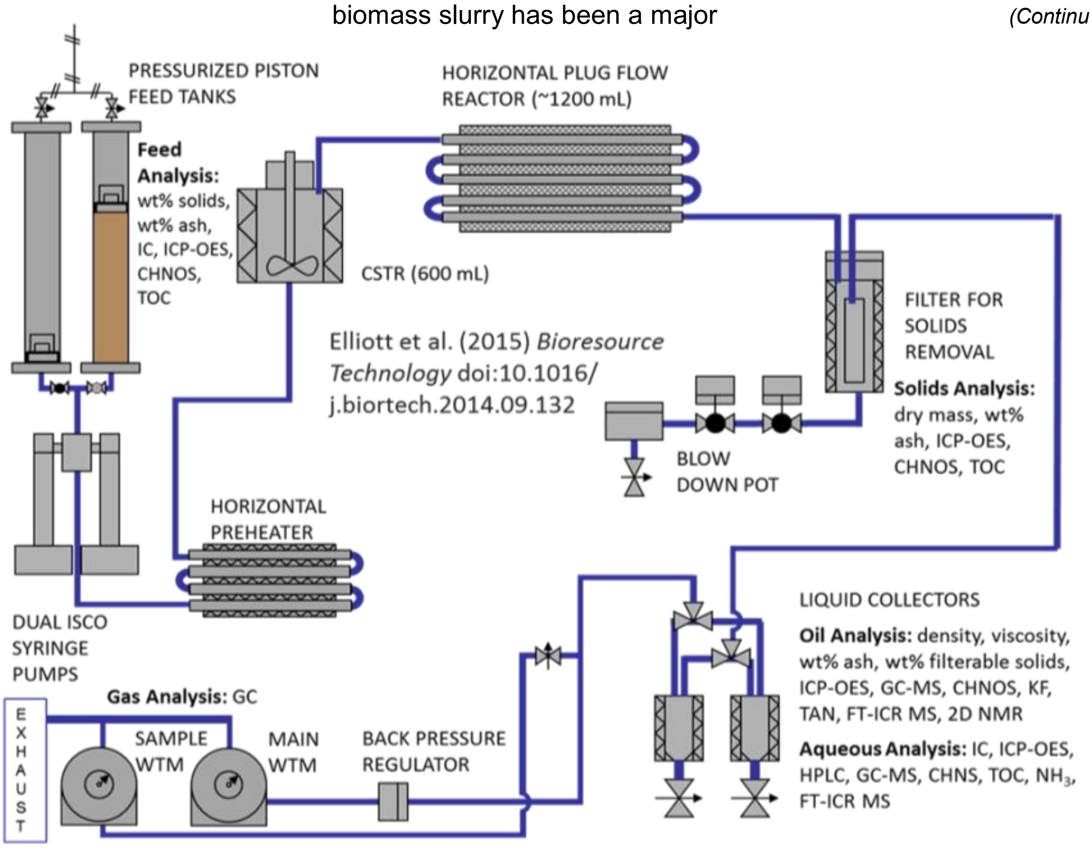

Elliott et al. (2015) *Bioresource Technology* doi:10.1016/j.biortech.2014.09.132

Figure 1: Bench-scale reactor system for HTL at PNNL.

Hydrothermal liquefaction of biomass *… continued*

Figure 2: HTL feedstock tested in 2015.

quality that could be achieved from a range of biomass feedstocks and derivatives. The project was completed and the final report issued.[3] Hydrothermal liquefaction research continued within the National Advanced Biofuels Consortium with the effort focused at PNNL. The bench-scale reactor was used for conversion of lignocellulosic biomass including pine forest residue and corn stover.[4] A complementary project was an international collaboration with Canada to investigate kelp (seaweed) as a biomass feedstock. The collaborative project included process testing of the kelp in hydrothermal liquefaction in the bench-scale unit at PNNL.[5] In addition, a major outcome of the recently completed consortium on algal biofuels (NAABB) was the development of HTL for whole microalgae processing to avoid the need for specialised algae species development and growth environments, as well as the processing for lipid recovery and biomass disposal as is typically described in the algae biofuels literature.[6]

Hydrothermal liquefaction at PNNL is performed in the hydrothermal processing bench-scale continuous-flow reactor systems. Slurries of biomass are prepared in the laboratory from whole ground biomass materials. Both wet processing and dry processing mills can be used, but the wet milling to final slurry is accomplished in a stirred ball mill filled with angle-cut stainless steel shot. The PNNL HTL system, as shown in Figure 1, is a continuous-flow system which has been evaluated including a one litre stirred tank preheater/reactor, a one litre tubular reactor, or other smaller bore tubular reactors, in various combinations. The product is filtered at high-pressure to remove mineral precipitate before it is collected and phase separated into the biocrude phase and the aqueous byproduct phase. The collection can be accomplished in the high-pressure collection system involving two vessels, which allow the liquid products to be collected batchwise and recovered either from the process flow or an online system for direct pressure letdown and recovery. The filter can be intermittently back-flushed as needed during the run to maintain operation. Byproduct gas is vented out of the wet test meter for volume measurement and samples are collected for gas chromatography compositional analysis.

Recent tests at PNNL have involved, in addition to micro-algae and macro-algae, other biomass types and wet waste biomasses, as shown in Figure 2.

The biocrude product is typically analysed for elemental content in order to calculate mass and elemental balances around the experiments. Detailed chemical analysis is performed by gas chromatography-mass spectrometry (GC-MS) and ^{13}C nuclear magnetic resonance (NMR) is used to evaluate functional group types in the biocrude. Sufficient product is produced to allow subsequent catalytic hydroprocessing to produce liquid hydrocarbon fuels.

The biocrude product from HTL is typically a more viscous product compared to fast pyrolysis bio-oil. There are several reasons for this difference. The HTL biocrude contains a lower level of oxygen because of the more extensive secondary reaction of the pyrolysis products. There are smaller amounts of the many light oxygenates derived from the carbohydrate structures as they have been further reacted to phenolic aldol condensation products. The biocrude is more hydrophobic because of the lower oxygen content and resulting lower polarity and therefore has a lower amount of dissolved water. Without the light oxygenates acting as solvents along with the water, the biocrude product is much more viscous. Related results are that the biocrude is less dense and has a higher energy content. These differences in properties led to the

(Continued on page 13)

Hydrothermal liquefaction of biomass … *continued*

earlier recognised and more recently validated belief that the HTL biocrude could be upgraded by catalytic hydroprocessing in a manner more similar to simple petroleum hydrotreating.

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Professor Michael J Antal Jr

Doug Elliott of Pacific Northwest National Laboratory, USA, remembers his friend and colleague, Professor Michael Antal, who passed away earlier this year


Professor Michael Antal, who passed away this year

Professor Michael Antal passed away on October 21st, 2015, at the age of 68. Michael Jerry Antal, Jr. graduated *Summa Cum Laude* with Highest Distinction in Physics and High Distinction in Mathematics from Dartmouth College in 1969. He earned an MS in Applied Physics in 1970, and a PhD in Applied Mathematics in 1973, both from Harvard University. Afterwards, Dr Antal spent two years as a theorist with the Thermonuclear Weapons Physics Group of the Los Alamos Scientific Laboratory, and six years with Princeton University. At Princeton he was a member of the faculty of the Mechanical and Aerospace Engineering Department and Director of the Renewable Resources Research Laboratory (R3L). He was one of the pioneers in thermochemical conversion of biomass, authoring the report on the subject, “Biomass Energy Enhancement,” for the President’s Council on Environmental Quality in 1978.

In 1981 Dr Antal was invited to assume the newly endowed Coral Industries Distinguished Professor of Renewable Energy Resources Chair with the University of Hawaii, where he was subsequently engaged in research on the pyrolysis of biomass materials. Dr Antal’s research interests included super-critical water catalytic gasification of biomass and high-pressure flash carbonisation of biomass for solid fuel production. During the period 2002-2005 he participated as a technical advisor in the NEDO-funded collaboration between Japan (Hiroshima University) and the Netherlands (Twente University), which focused on super-critical water gasification of biomass, helping to organise several of the workshops of international experts.

He was author or co-author of more than 100 peer-reviewed archival publications and 11 patents and patents pending. As of 2008 his citation record was in the top 1% of total citations in all fields of engineering.

He leaves a wide international circle of fellow researchers and colleagues in his field who were privileged both to work alongside him and to count him as a friend. A special issue of the journal Energy & Fuels is being organised to honour Professor Antal.

Biomass fast pyrolysis in Thailand

Adisak Pattiya of Mahasarakham University gives us an insight into ongoing work in Thailand

Biomass potential

Thailand is an agricultural country that has various biomass resources available for utilisation as summarised in Table 1. The agricultural residues in Thailand have an annual energy potential of about 25 million tonnes of oil equivalent (MTOE).

Fast pyrolysis units

Biomass fast pyrolysis research and development in Thailand is still in its infancy. Organisations involved in developing this technology are:

- Mahasarakham University (MSU);
- Thailand Institute of Scientific and Technological Research (TISTR);
- National Metal and Materials Technology Center (MTEC) of National Science and Technology Development Agency (NSTDA);
- Chulalongkorn University (CU)
- King Mongkut's University of Technology North Bangkok (KMUTNB);
- PTT Public Company Limited.

There are four types of fast pyrolysis reactors that have been developed in Thailand so far. These include four bubbling fluidised-bed reactors, two circulating fluidised bed reactors, two auger reactors and two free-fall reactors. Figures 1-3 show photos of the fast pyrolysis units, which have a biomass throughput capability ranging from 50g/h to 20kg/h. The largest fast pyrolysis unit in Thailand belongs to TISTR. It has been developed under the project "Innovation on Production and Automotive Utilisation of Biofuels from Non-food Biomass", which is a collaborative project of TISTR, NSTDA/MTEC and KMUTNB of Thailand and the National Institute of Advanced Industrial Science and Technology (AIST) and Waseda University of Japan. The original design was adapted from AIST and modified by TISTR researchers.[1]

Fast pyrolysis products

There are three products obtained from biomass fast pyrolysis: liquid bio-oil, solid char and non-condensable gases. The products are shown in Figure 4. In the laboratory fast pyrolysis unit the bio-oil can be fractionally condensed using a condensation unit. As can be seen from Figure 4, bio-oil can be fractionated into organic-rich liquid and an aqueous liquid fraction. Bio-oil can be used as a fuel for boilers, engines or gas turbines for generation of heat and

(Continued on page 15)

Table 1: Overview of biomass resources and their energy potential in Thailand.

Crop	Residue	Biomass availability (million tonne/year)	Higher heating value (MJ/kg)	Energy potential (million tonnes oil equivalent, MTOE)
Rice	Straw	15.7	12.3	4.6
	Husk	1.8	14.2	0.6
Sugarcane	Leaves/tops	27.7	15.5	10.2
	Bagasse	0.03	7.4	0.0
Cassava	Stalk	1.8	13.4	0.6
	Rhizome	13.2	5.5	1.7
Corn	Cob	0.3	16.6	0.1
Oil palm	Empty fruit bunch	1.3	7.2	0.2
	Frond	33.3	7.5	6.0
Rubber	Stalk/stem	0.7	14	0.2
Eucalyptus	Bark	6.0	6.3	0.9
			Total	**25.1**

Biomass fast pyrolysis in Thailand ...*continued*

Table 2: Yields and heating values of fast pyrolysis products.

Biomass	Yield (%, dry biomass basis)				Heating value (MJ/kg)	
	Bio-oil	Aqueous liquid	Char	Gas	Bio-oil	Char
Sawdust	58.1	9.3	18.4	14.2	20.0	24.4
Tung oil seed cake	56.4	17.6	14.7	11.3	29.9	15.5
Leucaena leucocepphala	46.2	18.4	17.1	18.3	20.3	20.0
Palm cake	44.6	16.5	29.5	9.4	27.1	20.0
Rice husk	42.6	10.7	30.7	16.0	15.4	21.0
Cassava rhizome	42.5	15.5	25.3	16.7	22.1	25.5
Cassava stalk	41.4	13.0	21.8	23.8	22.2	24.7
Eucalyptus bark	40.8	16.6	29.1	13.5	16.3	20.0
Jatropha seed cake	40.7	8.3	33.6	17.4	23.4	20.0
Sugarcane tops	35.0	16.0	30.5	18.5	20.0	22.8
Sugarcane leaves	33.9	12.0	26.5	27.6	21.7	21.9
Rice straw	31.5	15.5	30.7	22.3	18.5	22.0

electricity. Bio-oil can be upgraded either in the process or after production by, for example, the application of catalysts. An example of bio-oil produced from the catalytic pyrolysis is also shown in Figure 4. The appearance of the catalytic bio-oil is different from the typical one. It is oily and is separated from the water phase.

Twelve potential biomass samples were tested with MSU fast pyrolysis units in order to produce bio-oil, aqueous liquid, char and gas. The bio-oils were captured and condensed at room temperature with the aid of an electrostatic precipitator (ESP), whereas the aqueous liquids were obtained from low temperature condensers using dry-ice/acetone. The bio-oil yields shown in Table 2 are in the range of 31.5 - 58.1%. The maximum bio-oil yield was obtained with sawdust, whereas rice straw gave the lowest yield. The bio-oil higher heating value (HHV) is between 15-30MJ/kg with tung oil seed cake bio-oil giving the highest HHV. The char HHV is in the range of 15.5 - 25.5 MJ/kg.

(Continued on page 16)

Figure 1: Fluidised fast pyrolysis reactors in Thailand. *Clockwise from top left:* 50g/h MSU Bubbling Fluid Bed; 300g/h MSU Bubbling Fluid Bed; 3kg/h PTT Bubbling Fluid Bed; 100g/h MSU Circulating Fluid Bed; 20kg/h TISTR Circulating Fluid Bed; 50g/h MSU Bubbling Fluid Bed.

Biomass fast pyrolysis in Thailand *...continued*

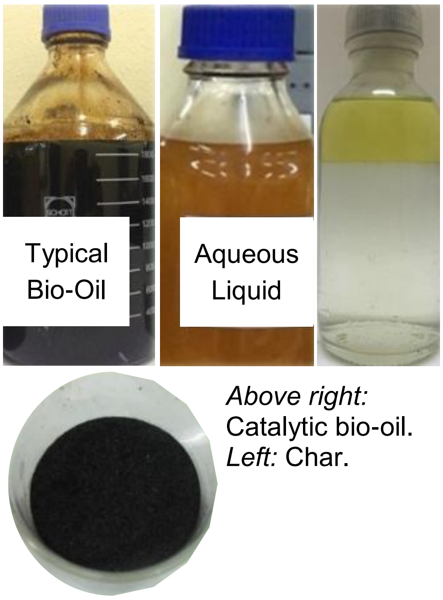
Figure 2: Screw fast pyrolysis reactors in Thailand.
Top: 300gh/MSU Twin Screw Reactor. *Bottom:* 3kg/h TISTR Extruded Screw Reactor.

Other properties of bio-oil produced in Thailand have also been tested and the results showed that the bio-oil met the international standard requirement for pyrolysis liquid according to ASTM D7544–12.

Conclusions

Current studies in Thailand have shown high potential for the technology as it can be used on virtually all types of available biomass. The future research and development of biomass fast pyrolysis in Thailand should be directed towards creating a pilot plant for production of both catalytic and non-catalytic bio-oil. Since biomass such as agricultural residues are not gathered in one place, shipping of the bulky solids to a large scale fast pyrolysis plant would not be economically feasible. Therefore, the development of a mobile fast pyrolysis plant in Thailand would be more appropriate provided that there are available applications accepting the bio-oil product as their fuels. Moreover, to enhance the economic feasibility of biofuel production from biomass by the fast pyrolysis process, the co-production of bio-char and other value-added specialty chemicals should be considered based on a biorefinery concept.

Figure 3: Free-fall fast pyrolysis reactors in Thailand.
Top: 100g/h MSU Free-Fall Reactor. *Bottom:* 200g/h MSU Free -Fall Reactor.



Above right: Catalytic bio-oil.
Left: Char.

Figure 4: Fast pyrolysis products.

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PYROCHEM: Biopolymers ^{13}C tracking during fast pyrolysis of biomass - A two-level mechanistic investigation

Marion Carrier (above) and Tony Bridgwater (below) of the European Bioenergy Research Institute, Aston University, UK, discuss exciting Marie Skłodowska-Curie research into fast pyrolysis

Figure 1: Closed-system climate chambers at IsoLife.

It has long been demonstrated that plant biomass can be efficiently converted into volatiles and limited amounts of gases and char by ways of a fast pyrolysis reactor. The mixture of aerosols/vapours is then rapidly quenched resulting in a relatively high yield of bio-oil, often as high as 70-75wt.% of the starting material on a dry basis.[1] In addition to the yield, the distribution of products determines their quality, and this is critically dependent on biomass type and its temperature-time history.[1] The pyrolytic liquid is a complex mixture of reactive organic compounds, which are usually categorised into functional families characterised by their properties such as acidity, distillability and oxygen content, etc.

The relative proportions of cellulose, hemicellulose and lignin in biomass feedstocks directly influence the chemical reactivity of biomass and have a significant impact on the composition of the bio-oil and its properties.[2] While it is still difficult to predict how the lignocellulosic composition impacts fast pyrolysis throughputs and the quality of its products, further insight in pyrolysis chemistry is required to (i) reveal key mechanistic details of the formation of bio-oil and (ii) define more precisely the interactions between lignocellulosic biopolymers.

To do this, myself and Professor Tony Bridgwater of the European Bioenergy Research Institute (EBRI) at Aston University, UK, have been recently granted a Marie Skłodowska-Curie Action named PYROCHEM. This project aims at reaching a new level of understanding of fast pyrolysis mechanisms by acquiring the necessary knowledge at a molecular-level using fractionation and isotopic characterisation techniques along with molecular dynamics calculations.

The application of these findings should contribute to the optimisation of the biomass fast pyrolysis process with the production of a more attractive pyrolysis-derived oil, thus providing an in-depth understanding of pyrolysis chemistry and supporting EBRI's current world-leading research in the field of catalysis and thermochemical conversion.

Support

Stable isotope labelled plant materials are currently provided by IsoLife (Wageningen, the Netherlands, www.isolife.eu).

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Simulating the in-reactor chemistry of fast pyrolysis and the application of products

Pelle Mellin of Swerea - Swedish Research, Sweden (previously of KTH Royal Institute of Technology) discusses his research

During 2011-2015 I carried out my PhD work at KTH Royal Institute of Technology. My work[1] focused on modelling in contrast to the previous experimental efforts[2] at the Division of Energy and Furnace Technology (now called Unit of Processes) at the department of Materials Science.

Owing to previous numerical modelling experiences of shaft furnaces used in metallurgical processes, I decided to take on the challenge of modelling (CFD), the dynamics of the bubbling fluidised bed reactor-coupled with the chemistry of the biomass decomposition by Computation Fluid Dynamics.

A two-step approach was used to model the primary pyrolysis and secondary pyrolysis separately. A factor which was investigated by modelling was the atmosphere in the pyrolysis reactor, in order to understand more about the process and underlying chemistry. The physical effects of the atmospheres were considerable, although surprisingly more so in the case of the secondary pyrolysis. The atmosphere affected the distribution of primary pyrolysis in the bed zone (Figure 2) but not the decomposition-pathways of the different components. Overall, for secondary pyrolysis the atmosphere affected residence time strongly and also the temperature profile in the fluidised bed reactor system (Figure 3).

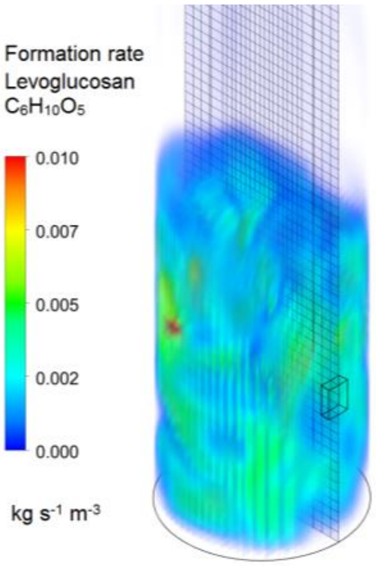

Figure 1: In-silico formation of Levoglucosan, in a lab-scale Bubbling Fluidised Bed (BFB).

The chemical effect (see Figure 4) of the atmospheres could also be investigated thanks to a chemistry model developed by a team at the Politecnico di Milano, led by Professor Eliseo Ranzi. This chemistry model (available at creckmodeling.chem.polimi.it) of secondary pyrolysis was implemented in CFD. More specifically, a Probability Density Function (PDF) framework was used alongside several acceleration techniques such as Dimension Reduction, Chemistry Agglomeration and *in situ* Tabulation (ISAT).

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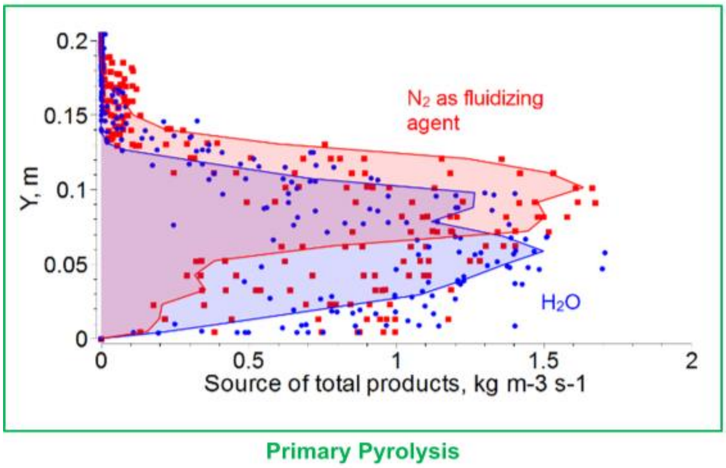

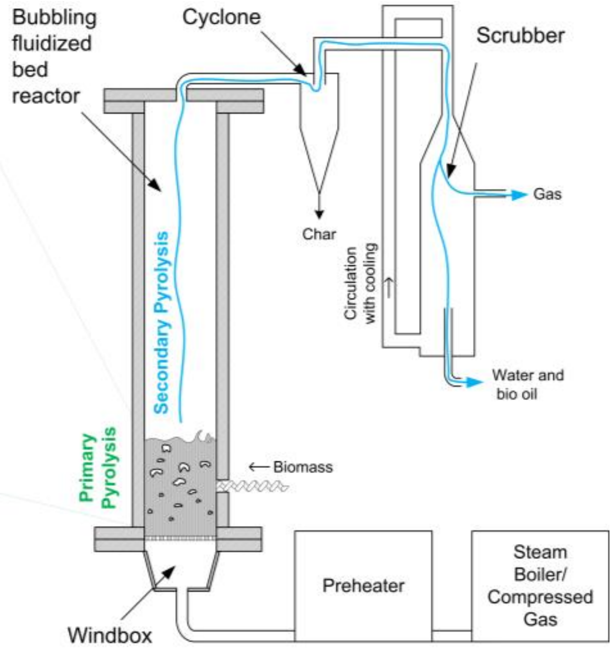

Figure 2: *Above:* Effect of atmosphere during Primary Pyrolysis, along the height of the bed zone.[3]
Right: Layout of the lab-scale Bubbling Fluidised Bed system, with Primary Pyrolysis and Secondary Pyrolysis indicated.

Simulating the in-reactor chemistry
...continued

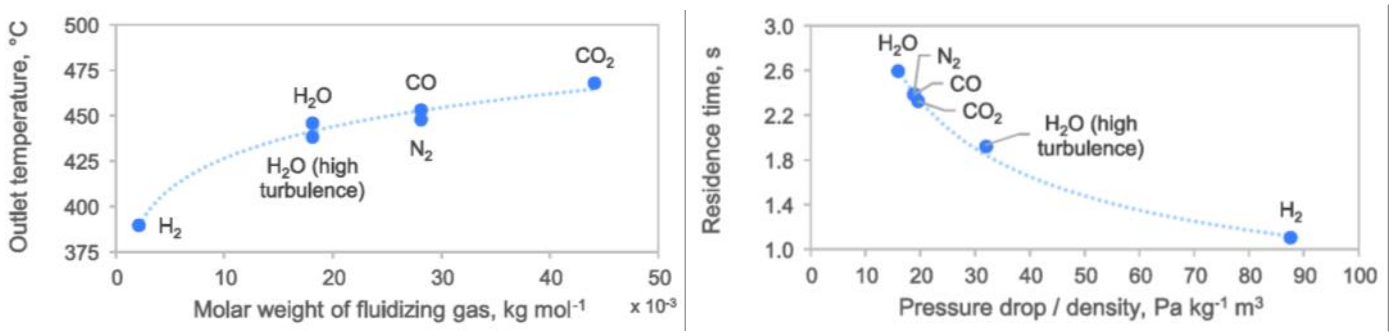

Figure 3: Physical effect of atmosphere during secondary pyrolysis, temperature (left) and vapour residence time (right).[4]

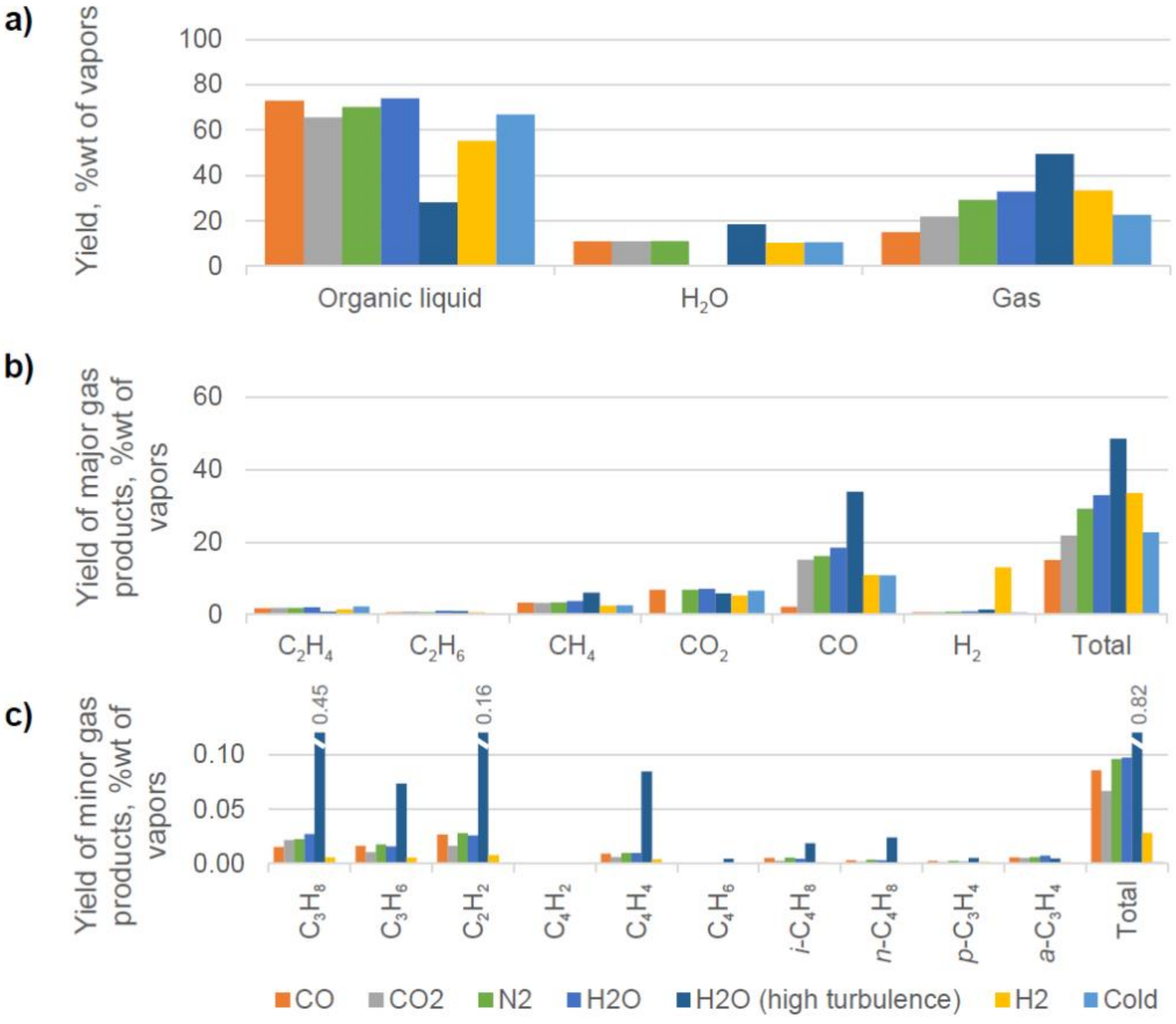

Figure 4: Chemical effect of atmosphere on (*top*) overall yields and yields of noncondensable gases (*middle*) showing major compounds and (*bottom*) minor compounds.[3]

Simulating the in-reactor chemistry ...continued

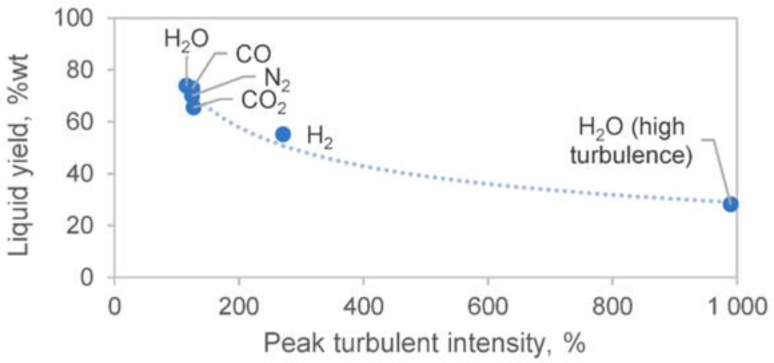

Figure 5: Effect of atmosphere on turbulent intensity, and in turn the liquid yield.[3]

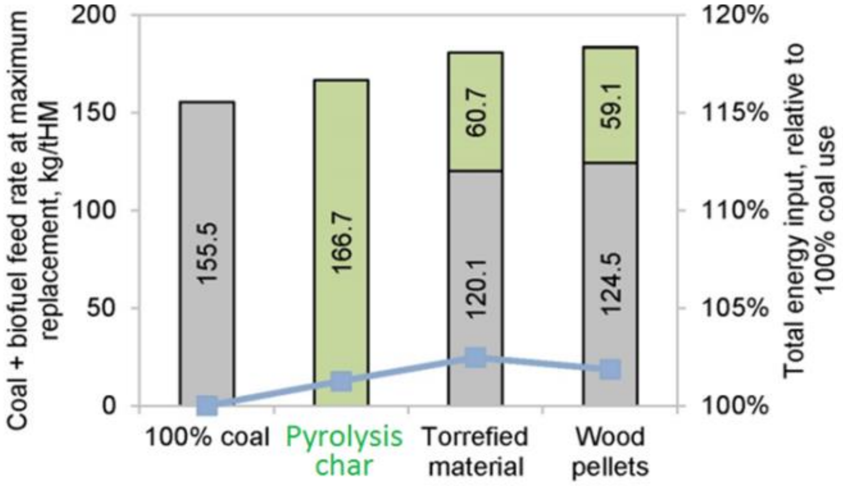

Figure 6: In the blast furnace process: replacement potential of pyrolysis char vs torrefied material and raw wood pellets. Pyrolysis char can replace coal completely.[5]

An advantage of this integration is that turbulence-interaction with the chemistry can be studied; here the atmospheres affect the turbulent intensity which the primary vapours encounter in the reactor. The model actually suggested that turbulence was an important driver for secondary pyrolysis (see Figure 5), more so than the temperature and residence time, which were also affected by the atmosphere.

I also took on work around pyrolysis applications, more specifically the use of pyrolytic products in blast furnaces. In blast furnaces, an auxiliary fuel (or injectant) is commonly used which covers about one-quarter of the total carbon-need of the blast furnace. This auxiliary fuel (normally coal) provides heat in the mid-section of the furnace and since it is simply combusted without actually contributing to the reduction, the requirements on the properties are less demanding. Here, pyrolysis char can play an important role since it has been found that pyrolysis char can completely replace coal and therefore contribute to lowering emissions from the steel industry (see Figure 6).

During the course of my PhD studies at KTH, I visited Aston University, UK, as a BRISK student. KIC Innoenergy also supported the visit through the PhD School. I now work with Process Development for Powder Materials and Additive Manufacturing at Swerea - Swedish Research at their KIMAB site, located in Kista, Stockholm.

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Enabling innovations via thorough characterisation of pyrolysis oil

Linda Sandström (above) and Magnus Marklund (below) of SP Energy Technology Centre AB, Sweden, discuss work motivated by the SWEPOIL initiative

Figure 1: The pilot scale cyclone pyrolyser at SP ETC.[1]

Pyrolysis oil is a very complex substance, and physical analyses to cover most of the constituents in the oil are difficult and time consuming. Analysis and characterisation are, however, very important to enable further development and innovations in pyrolysis processes, especially those related to the upgrading and use of the pyrolysis oil. Motivated by the SWEPOIL initiative (which aims to gather and share knowledge around pyrolysis oil in Sweden), a research project has recently been initiated at SP Energy Technology Centre (SP ETC). The project focuses on developing competence related to the analysis and characterisation of pyrolysis oil by performing more fundamental characterisation, such as titration of water and acids, as well as detailed and state of the art analyses by advanced instrumentation.

The pilot scale cyclone reactor at SP ETC (see Figure 1), has been used to produce pyrolysis oil with a nominal feed rate of 20kg/h to the pyrolyser.[1] In the first part of the work a number of raw materials have been pyrolysed, including reed canary grass, forest residue and stem wood from pine and spruce.

(Continued on page 22)

Enabling innovations
...continued

Figure 2: A Shimadzu GCMS QP-2010 Ultra equipped with ZOEX liquid nitrogen modulation.

In order to achieve a more fundamental understanding of the pyrolysis process and of the properties of the oil, deep insight into the chemical composition of the oil is needed. Therefore, a comprehensive two-dimensional gas chromatography (GCxGC) mass spectrometry (MS) instrument has been installed at SP ETC during the autumn of 2015 (see Figure 2). Comprehensive GCxGC (also denoted 2D GC) is a relatively new technique which offers significantly enhanced resolution and identification of organic analytes in complex mixtures compared to one-dimensional GC.[2] In comprehensive GCxGC, the entire sample is separated by two columns with complementing selectivity (e.g. a polar and a non-polar column).

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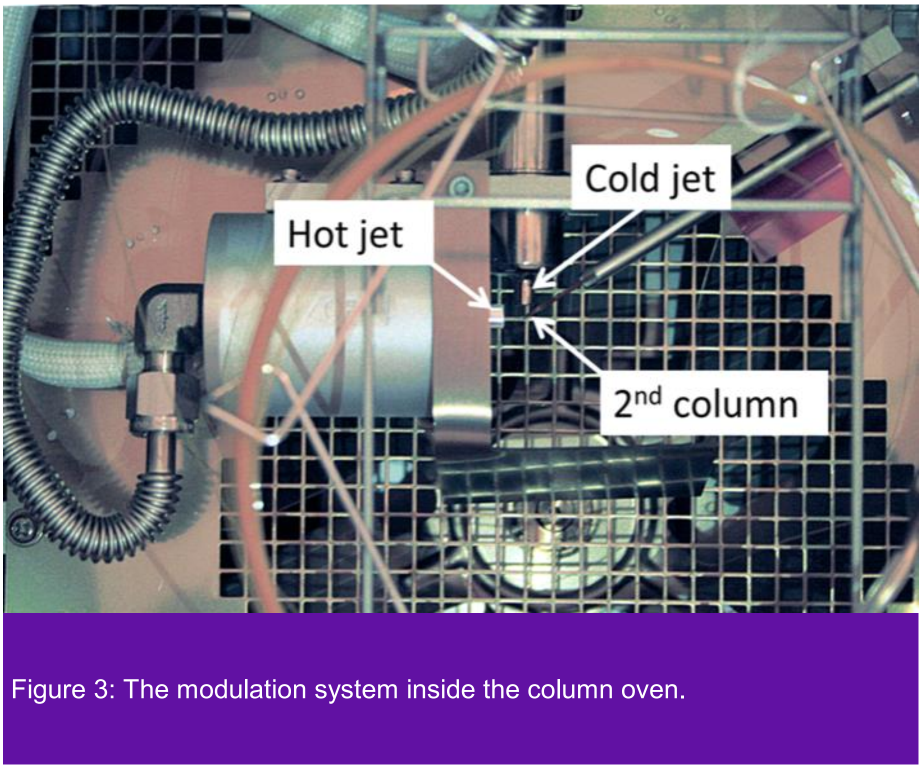

Figure 3: The modulation system inside the column oven.

Enabling innovations
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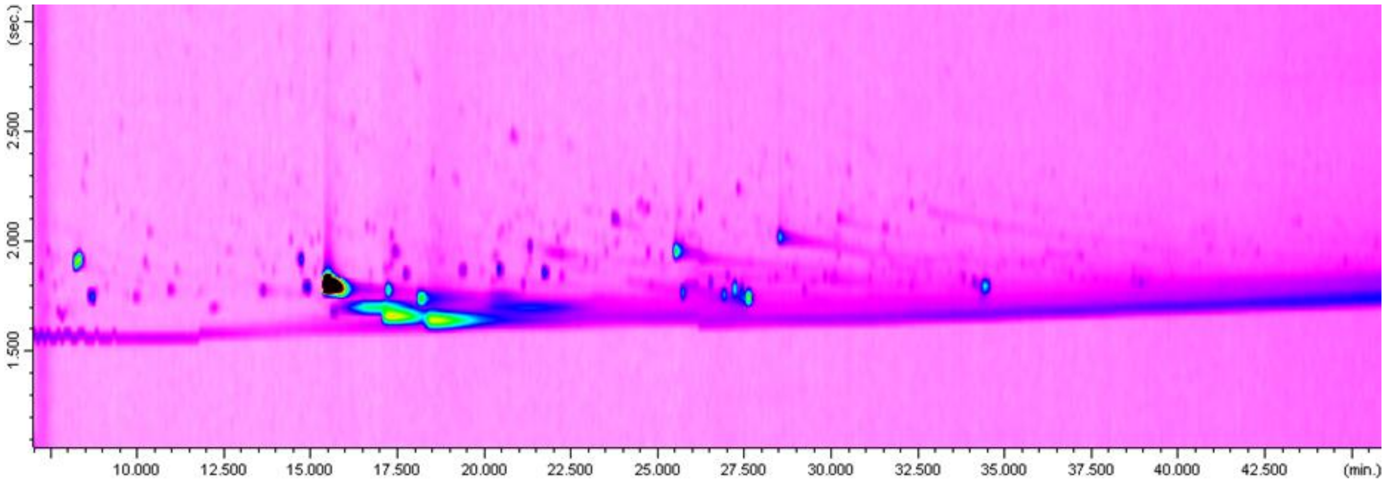

Figure 4: 2D-chromatogram of pyrolysis oil from stem wood (pine and spruce).

The 2D-modulation consists of a cold jet and a hot jet (see Figure 3). The cold jet is cooled by liquid nitrogen, and is used to freeze the outlet of the first column. At determined intervals, e.g. 6s, the hot jet is activated which releases the frozen components, thereby injecting them to the second column. The total residence time in the second column is very short, and while the injected components are eluted through the second column, the outlet of the first column is again frozen by the cold jet.

Pyrolysis oil from the cyclone pyrolyser has been analysed by comprehensive GCxGC-MS (see Figure 4). The chromatogram was recorded on a Shimadzu GCMS QP-2010 Ultra using ZOEX liquid nitrogen modulation. The MS is of quadrupole type. The first column was a 30 m SLB-IL61 (Supelco) and the second was a 1 m Rxi-1ms (Restek), and the sample was diluted in acetone. The choice of column set is a very important parameter in method optimisation, and results from both non-polar-polar combinations[3] and the opposite (so called reverse phase column combination)[4] have been reported for the analysis of pyrolysis oil.

The benefit of the second dimension can be seen in Figure 4, as not all components are separated in the first dimension (along the x-axis). Examples of identified components include acids, aldehydes, ketones and phenols, and work is currently ongoing to verify identification and to optimise the analysis conditions. Quantification of important compounds by the FID (flame ionisation detector) connected to the GCxGC is also to be performed. In order to enable solids or heavy parts of the pyrolysis oil to be studied, the GCxGC-MS is also equipped with a single step pyrolyser.

The GCxGC-MS instrument is anticipated to be very valuable for the research related to the pilot scale cyclone reactor, especially for understanding the possibilities for upgrading and use of the oil, and research is ongoing.

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Catalytic pyrolysis and bio-oil upgrading research at Heriot-Watt University

Aimaro Sanna describes ongoing research at Heriot-Watt University, Scotland, UK

Heriot-Watt University (HWU) has been actively researching the production of partially deoxygenated pyrolysis bio-oils through the incorporation of heterogeneous catalysts in the pyrolysis process. Three research teams at Heriot-Watt are developing catalytic systems for the production of inexpensive renewable liquid fuel and commodity chemicals out of renewable products such as microalgae, wood and organic wastes, in collaboration with international researchers. These teams are led by myself, Dr John Andresen and Professor Raffaella Ocone.

My own team and Dr Andresen's team are part of the EPSRC funded Centre for Innovation in Carbon Capture and Storage (CICCS) based at the School of Engineering and Physical Science (EPS), which addresses issues of global importance in the area of sustainable and affordable energy technologies. Professor Ocone is a member of the UK Biochar Research Centre funded by the Leverhulme Trust. The teams utilise fixed bed reactors for catalyst screening, a 100kg/h bubbling fluidised bed pyrolysis system to study *in situ* catalytic upgrading, and a continuous pyrolysis unit with a biomass capacity of 2kg/h to process waste polypropylene.

My recent research has focused on the development of metal oxide and zeolite catalysts towards the production of more stable and upgradable pyrolysis oils. Microalgae have been targeted due to their potential in terms of high photosynthetic efficiency, high productivity, high lipid content (up to 80%) and the absence of competition with food crops. Although microalgae to biodiesel has been studied extensively, microalgae catalytic pyrolysis has received less attention. Recent studies suggest that the whole pyrolysis of microalgae grown in waste effluents is an attractive technology in terms of greenhouse gas emission and energy consumption.[1] My team is developing catalysts to improve the properties of the bio-oil and selectively control the distribution towards aliphatic and aromatic products. Among the tested catalysts, Ni–Ce/Al_2O_3 was able to retain 59% of the algae starting energy in the bio-oil (compared to only 41% in the absence of the catalyst), lower the content of acids, improve the C/O molar ratio from 3.6 (without catalyst) to 5.1, and produce a bio-oil rich in aliphatics.[2] CeO_2 as such is instead particularly good for producing ketones, while metal doped zeolites are being developed for maximising the production of aromatics. There are still research barriers to overcome for the catalytic conversion of algal biomass, including that deoxygenation results in C losses in coke formation due to removal of hydrogen from the algal biomass. Another limitation is represented by algae's intrinsic high N-content which can produce NOx if combusted, and by the high ash content that can lead to catalyst deactivation.

Professor Ocone's pyrolysis research is focused on comparing fast and slow pyrolysis with the intent of recovering valuable products primarily from biomass. Pyrolysis of plastic wastes is also studied as an alternative to landfill. The work is carried out in collaboration with the Institute for Chemicals and Fuels from Alternative Resources (ICFAR) at Western University in the US, the University of Saskatchewan and McGill University in Canada, and the University of Edinburgh in the UK. The specific work at HWU involves the modelling of the kinetics of pyrolysis, aiming towards optimising process conditions to achieve high yields of high-added value products such as phenols. HWU has developed a novel lumping methodology which has been shown to be successful in predicting the yields of pyrolysis of lignin and plastic wastes. The process conditions are also investigated and the models developed are validated against experiments carried out at ICFAR and the University of Edinburgh. The production of biochar is investigated with the aim of activating the solid residue and implementing it in a number of applications such as adsorbents.

Dr Andresen's pyrolysis work is currently focused on conversion of plastic into value-added chemicals. Consumption of plastics, in particular high density polyethylene (HDPE), has increased 8% each year since the 1950s with a projection of reaching 300 million tonnes by 2015, where more than 50% is discarded after a single use. As a result, several countries, such as Scotland, have long-term goals of "zero waste". Scotland aims to recycle 70% of all waste by 2025 with only 5% of waste going to landfill, as policies recommended to the Scottish Government by Zero Waste Scotland include a ban on disposal

(Continued on page 25)

Catalytic pyrolysis and bio-oil upgrading ...continued

of collected dry recyclables to landfill or incineration. Due to the mixed nature of collected plastics, recycling through pyrolysis back into a chemical feedstock is key. Therefore, Dr Andresen's group focuses on catalytic pyrolysis of plastics not suitable for sorting into non-aromatic liquids. In particular, modified sulphated zirconia has been found to achieve close to 100% plastic conversion with a liquid yield close to 70wt%. The liquid contains mainly paraffinic, olefinic and naphthenic compounds in the gasoline and kerosene boiling point range. The non-aromatic conversion pathway and high liquid yield was found to be strongly linked with a modification of the acidic sites of the catalysts. Work is being developed on the potential to integrate this within existing refinery settings.

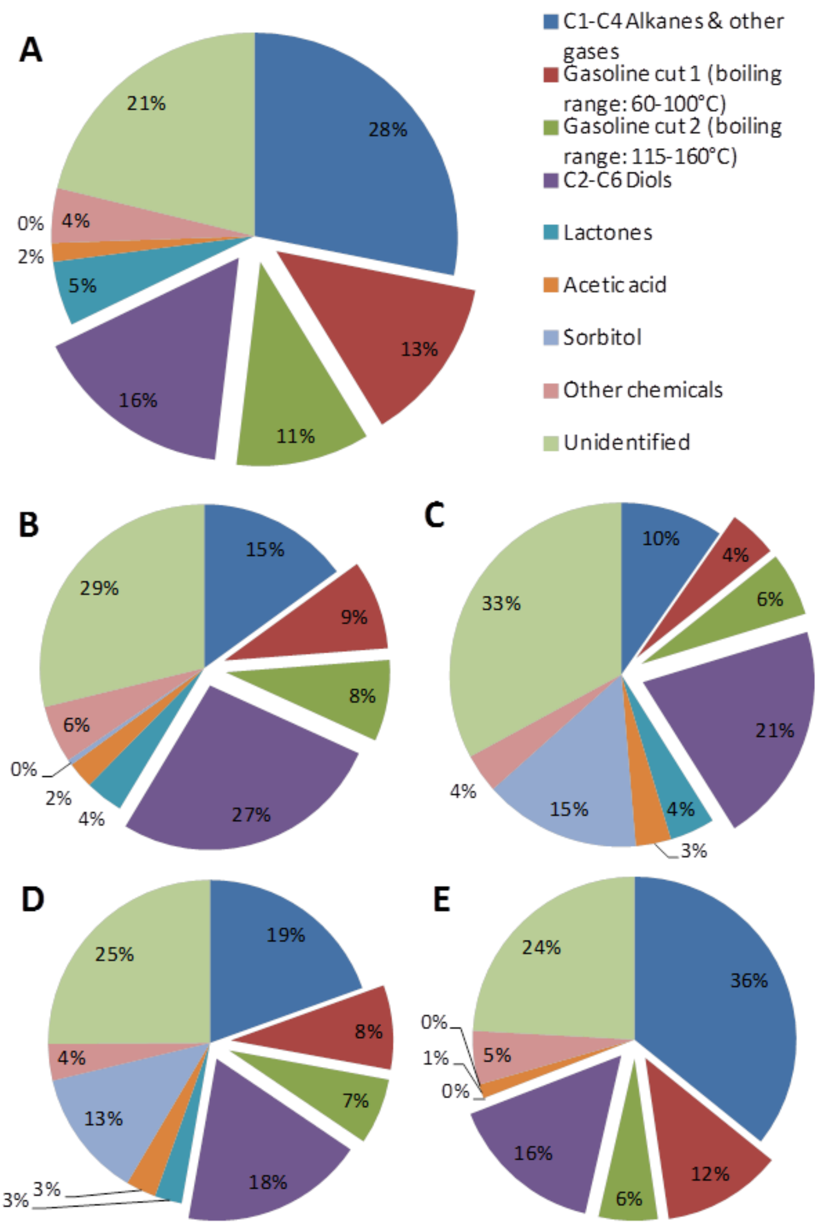

Figure 1: Product selectivity in single and two-stage hydrogenation of aqueous fraction of bio-oil A: Ru/C-Pt/C, 125-250°C, 750 psi, B: Ru/C-Pt/C, 125-250°C, 1450 psi, C: Ru/C-Pt/C, 125-220°C, 1450 psi, D: Ru/C-Pt/C, 125-220°C, 750 psi, E: Ru/C-Pt/C, 125-275°C, 1450 psi.[4]

My own team is also working in collaboration with Professor George W Huber (University of Wisconsin-Madison, US) to show how bio-oils produced from biomass can be upgraded into high commodity chemicals such as mono-alcohols, diols, light olefins and aromatic hydrocarbons which are used in the production of plastics.[3,4] Using cutting-edge technology, lignocellulosic bio-oils have been converted into 11 different biomass-derived feedstocks using integrated hydroprocessing with a zeolite catalyst that makes them more compatible with current fuels and chemical crude oil refinery settings.[3] This is a promising and flexible integrated catalytic conversion pathway that would sensibly decrease the economic disadvantage of biomass compared with fossil fuels and would make possible the conversion of biomass on an industrial scale.[3,4] Oxygenated gasoline additives, alcohols, and diols can be produced by increasing the intrinsic hydrogen content of bio-oil in a two-stage continuous hydrogenation process (Figure 1). A Low Temperature Hydrogenation (LTH) step converts the aldehydes, ketones, and sugars in bio-oil to their corresponding alcohols with a Ru catalyst. The alcohols are thermally stable and can be further converted into the desired products by High Temperature Hydrogenation (HTH). Homogeneous coke formation is minimised at 125°C, at which the bio-oil is successfully hydrogenated to EG, PG and sorbitol with only 7% carbon loss to gas and solid phase products. No sign of deactivation was observed in LTH after 80 hours' time on stream. A HTH step then converts sorbitol to products including diols and monohydric alcohols. Hydrogenolysis and secondary hydrogenation reactions are predominant in the HTH step (Figure 2). Platinum was found to

(Continued on page 26)

Catalytic pyrolysis and bio-oil upgrading ...continued

be a suitable catalyst for HTH. Up to 45% of the starting carbon was successfully converted to gasoline blendstocks and C2 to C6 diols (Figure 1). The product distribution from the HTH can be controlled using the pressure and second stage temperature. High yields of gasoline blendstocks can be obtained by operating at low total pressure or at high second stage temperature (e.g. 275 °C). High pressure in the two-stage process minimises carbon loss to gas phase.[4]

Future advances in the field of metal and zeolite catalysts, combined with reaction engineering, will lead to the design of even more efficient and economical processes to convert biomass and waste resources to renewable chemical industry feedstocks and biofuels.

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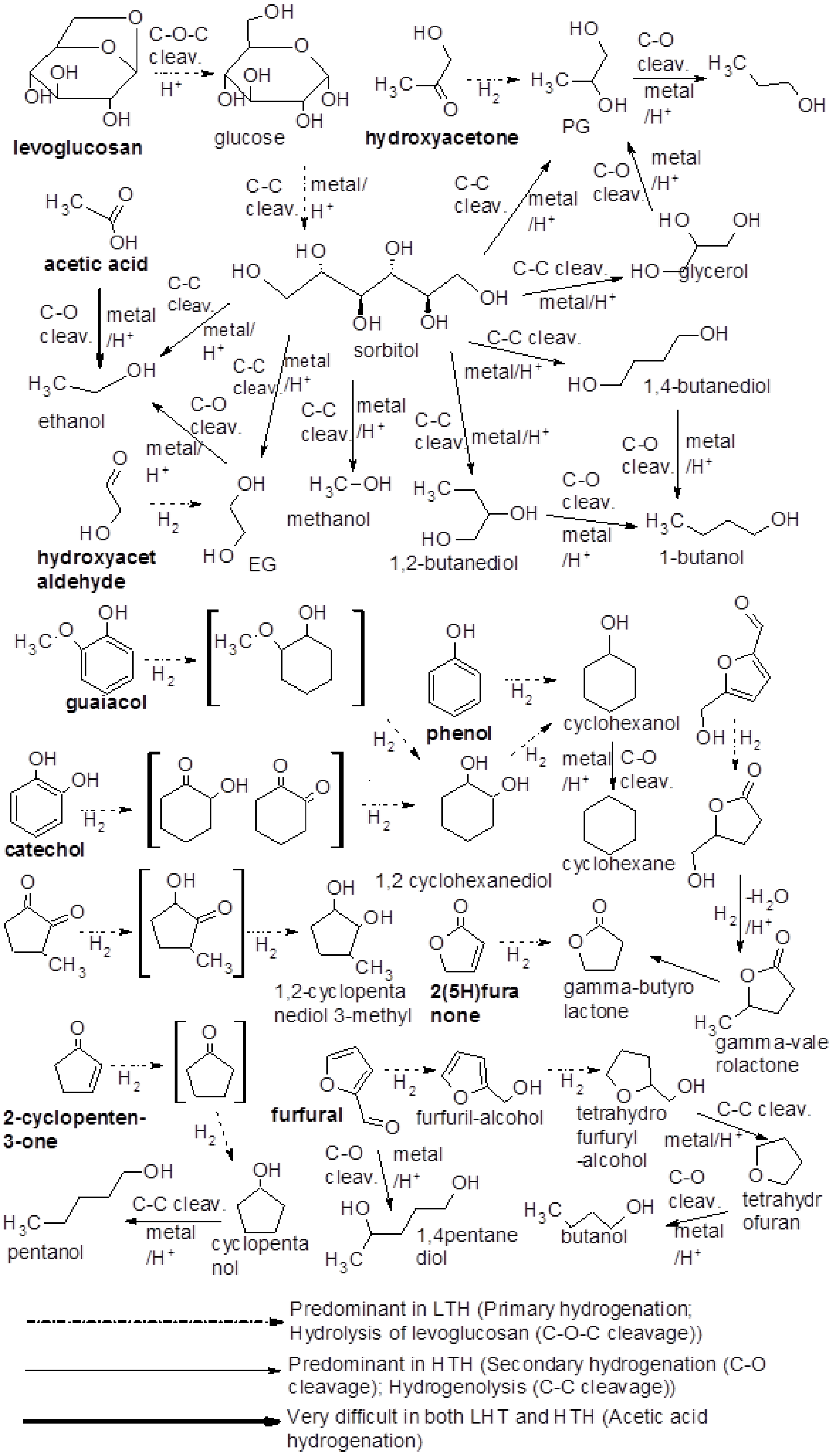
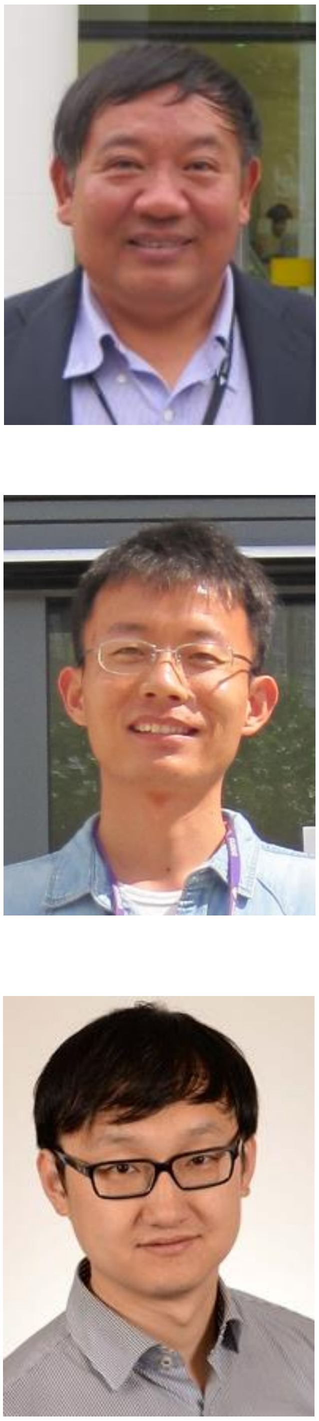


Figure 2: Reactions network involved in the conversion of bio-oil. Reactants' names in bold, [ ] intermediate compounds.[4]

References:
1. Robert M. Handler, David R. Shonnard, Tom N. Kalnes, F. Stephen Lupton (2014), Life cycle assessment of algal biofuels: Influence of feedstock cultivation systems and conversion platforms, Algal Research, 4,105-115.
2. T Aysu, A Sanna (2015), Nannochloropsis algae pyrolysis with ceria-based catalysts for production of high-quality bio-oils, Bioresource Technology, 194, 108-116.
3. TP Vispute, H Zhang, A Sanna, R Xiao and GW Huber (2010), Renewable Chemical Commodity Feedstocks from Integrated Catalytic Processing of Pyrolysis Oils, Science, 330, 6008, 1222-1227.
4. A Sanna, TP Vispute, GW Huber (2015), Hydrogenation of the Aqueous Fraction of Bio-oil with Ru/C and Pt/C Catalysts, Applied Catalysis B: Environmental, 165, 446-456.

Commercial scale biomass fast pyrolysis plant built in China

Ronghou Liu and Wenfai Cai, both of Shanghai Jiao Tong University, China, and Yang Yang of Aston University, UK, describe fast pyrolysis developments in China



Ronghou Liu (top), Wenfai Cai (middle), and Yang Yang (bottom)

As one of the most populated countries in the world, China is facing energy and environmental pressures and challenges from the rapid development of the international economy. Traditional methods to deal with agricultural waste and landfill can no longer meet the country's environmental or energy needs. It is therefore important to develop large-scale renewable energy plants.

A project entitled "Development of Equipment for Biomass Fast Pyrolysis for Bio-oil Production and its Demonstration in Thousand Tonne Scale" was supported by the Ministry of Science and Technology of China between 2011 and 2013. The project was coordinated by Shanghai JiaoTong University, with other partners including Zhejiang University, Shandong University of Technology, Guangzhou Institute of Energy Conversion, Chinese Academy of Science, University of Science and Technology of China, University of Science and Technology of South China, Liaoyang Hengxing Company Ltd, and Shaanxi Yingjiliang Biomass Company. Professor Ronghou Liu from Shanghai JiaoTong University was Project Coordinator, and this project led to many successes in the field of biomass pyrolysis.

During the project Shanghai JiaoTong University worked with

(Continued on page 28)

Figure 1: Ronghou Liu and colleagues with the project sign.

Commercial scale biomass fast pyrolysis ...*continued*

Figure 2: Main reactor at the Tongchuan plant.

Shaanxi Yingjiliang Company in developing and building a large scale biomass fast pyrolysis plant in the rural area around Weinan City, alongside its other plant in Tonchuan City, both in Shaanxi province. Shaanxi Yingjiliang's plants consist of a total of three 3000 kg/h circulating fluidised bed pyrolysis systems with a total investment value of 166 million CNY (USD 26.1 million), and Shaanxi Yingjiliang lays claim to the largest commercial fast pyrolysis plant in China. The Tongchuan plant was established in 2007 and its fast pyrolysis system has been in operation since 2011. The Weinan plant was established in July 2012. and started operating in May 2013, with the second system following in June 2014. The two plants have a total productivity of 30,000 tonnes of bio-oil and 20,000 tonnes of char per year from agricultural residues. The Weinan plant is currently dormant due to technical issues and the Tongchuan plant is currently operational. The fast pyrolysis systems consist of 5 main parts: feeding and pre-heating system, reactor and bed material circulation system (with pyrolysis gas loop), cyclones, quench and condensation system, and char separation system. The bio-oil from rice husks generally contains about 25% moisture and has a heating value around 20MJ/kg. The pyrolysis systems can work for approximately 7500 hours every year and requires roughly 50 days per year to give the system a break and to undertake maintenance work.

The project successfully solved the problems relating to large scale fast pyrolysis of biomass and continues to provide a valuable source of reference and technical support for biomass fast pyrolysis and potential bio-oil utilisation. The bio-oil from Yingjiliang's plants has a good local market value due to its good quality and relatively low price compared to heavy fuel oil. The bio-oil is primarily sold to local heat suppliers as fuel for boilers. In Shaanxi province, the local government has banned burning cheap low quality coal which is causing severe air pollution and encourages local heat suppliers to purchase bio-oil for heat production. The production cost of rice husk derived bio-oil is about 1800 CNY per tonne but it can be sold for at least 3300 CNY per tonne in the local fuel market. Each fast pyrolysis system is estimated to make about 5 million CNY profit annually from selling bio-oil and char and brings an additional 1 million CNY subsidy for local government.

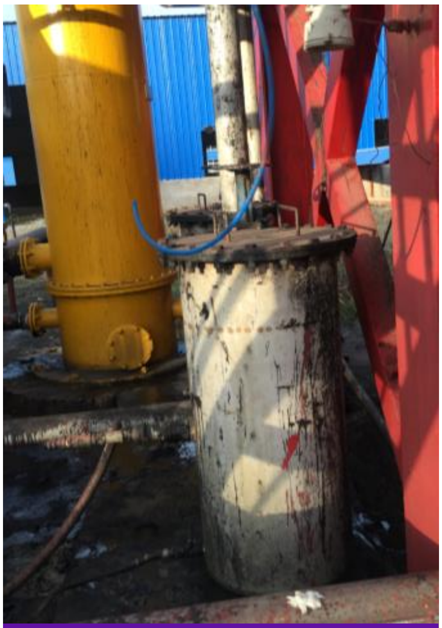


Figure 3: Oil collecting vessel.

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tcbiomass2015

Doug Elliott of Pacific Northwest National Laboratory, USA, gives a rundown on the tcbiomass2015 conference

tcbiomass2015 - the fourth international conference on thermochemical biomass conversion science - presented the latest research and innovations in the exciting field of bioenergy for the further deployment of clean energy technologies. Participants engaged in three days of technological interchange with the world's leading researchers, scientists and engineers in biomass thermochemical conversion, with topics ranging from gasification and pyrolysis to hydrothermal processing and upgrading. The conference was held in Chicago at the Westin Chicago River North Hotel on 2-5 November 2015, and over 280 attendees provided an energetic and engaging meeting. The participants included senior experts and young researchers, major corporations and entrepreneurial startups, academics and plant operators. As in the past, tcbiomass2015 was organised by GTI under the leadership of Terry Marker and the scientific panel, with Vann Bush and Kyriakos Maniatis serving as conference co-chairs. The conference agenda included 5 keynote speakers:

- Adam Brown - "The Role of Bioenergy in IEA's Mid-Term Market Report 2015";
- Y.B. Ramakrishna - "Biomass to Fuel/Energy—Opportunities and Challenges in India";
- Dr Jonathan Male - "US Department of Energy Perspectives on Renewable Energy";
- Gerhard Muggen - "First Production Results of Pyrolysis Oil Production at EMPYRO: Results of the Oil Firing and Future Commercial Applications in Refineries";
- Dr Jennifer Holmgren - "Industrial Biotechnology: The Pathway to a Carbon Smart Future".

Sixty-four technical presentations were organised into plenary and parallel sessions including:

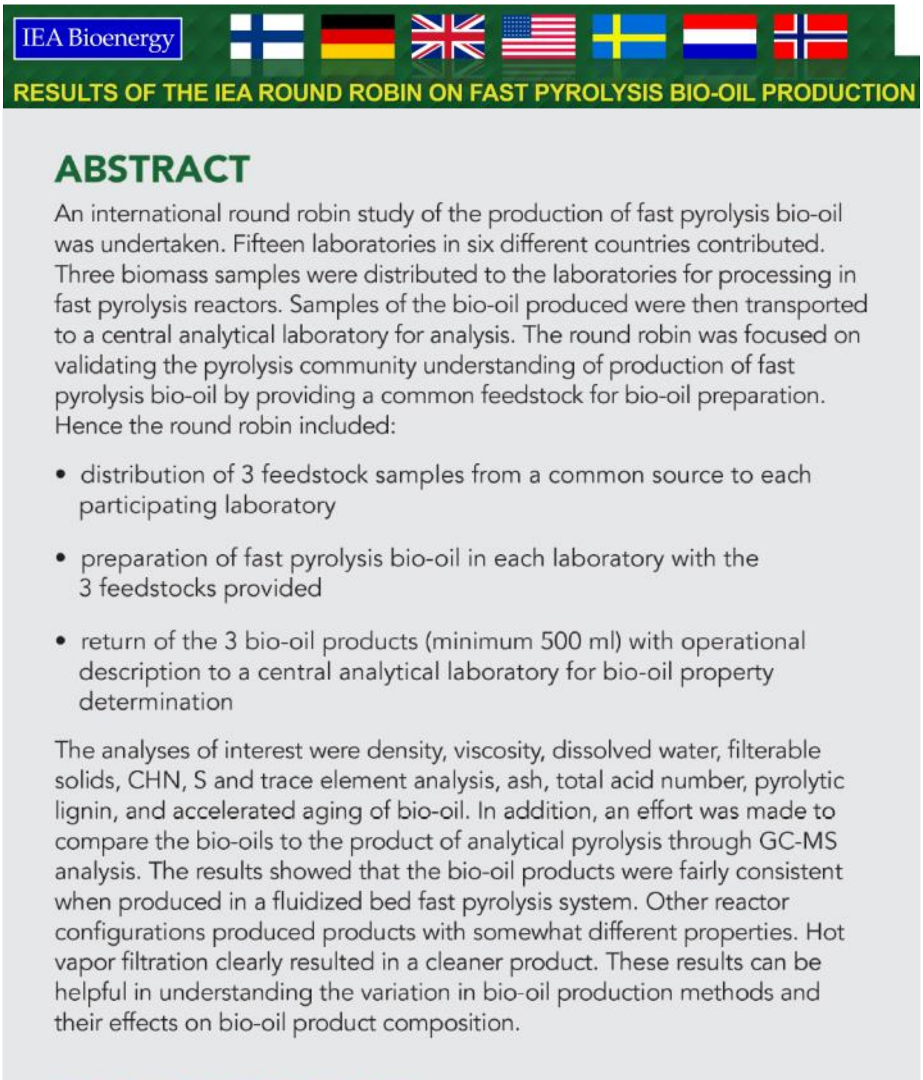
- Gasification (1.5 sessions including 17 presentations);
- Pyrolysis (2 sessions including 28 presentations);
- Upgrading (1 session including 14 presentations);
- Liquefaction (half session including 5 presentations).

The conference also included 115 poster presentations in the same topic areas. Included in the collection was one prepared by Task 34 describing some of the results of the recent Round Robin

(Continued on page 30)

Figure 1: View from the conference refreshments area.

tcbiomass2015 *...continued*



RESULTS OF THE IEA ROUND ROBIN ON FAST PYROLYSIS BIO-OIL PRODUCTION

ABSTRACT

An international round robin study of the production of fast pyrolysis bio-oil was undertaken. Fifteen laboratories in six different countries contributed. Three biomass samples were distributed to the laboratories for processing in fast pyrolysis reactors. Samples of the bio-oil produced were then transported to a central analytical laboratory for analysis. The round robin was focused on validating the pyrolysis community understanding of production of fast pyrolysis bio-oil by providing a common feedstock for bio-oil preparation. Hence the round robin included:

- distribution of 3 feedstock samples from a common source to each participating laboratory
- preparation of fast pyrolysis bio-oil in each laboratory with the 3 feedstocks provided
- return of the 3 bio-oil products (minimum 500 ml) with operational description to a central analytical laboratory for bio-oil property determination

The analyses of interest were density, viscosity, dissolved water, filterable solids, CHN, S and trace element analysis, ash, total acid number, pyrolytic lignin, and accelerated aging of bio-oil. In addition, an effort was made to compare the bio-oils to the product of analytical pyrolysis through GC-MS analysis. The results showed that the bio-oil products were fairly consistent when produced in a fluidized bed fast pyrolysis system. Other reactor configurations produced products with somewhat different properties. Hot vapor filtration clearly resulted in a cleaner product. These results can be helpful in understanding the variation in bio-oil production methods and their effects on bio-oil product composition.

Figure 2: Abstract element of the poster presentation prepared by Task 34 describing some of the results of the recent Round Robin on bio-oil production. Click here to view the full version alongside other pyrolysis-related posters featured at tcbiomass.

on bio-oil production (see Figure 1). Presentations and many of the posters are available on the conference website or via clicking here.

The attendees at the conference represented 22 countries, with 31% of delegates coming from outside the US. The attendees came from a variety of backgrounds, with 35% from universities, 32% from research institutes, 27% from industry and 6% from government.

The conference was reported as a great success and one of the many highlights was the presentation of the Don Klass award, which was awarded to Professor Robert Brown of Iowa State University for Excellence in Thermochemical Conversion Science to recognise his leadership in shaping the agenda of tcbiomass and his educational contributions to the field.

tcbiomass2015 hosted a competitive student poster contest to help connect today's top engineering and science students with global bioenergy experts. 44 poster presentations were evaluated throughout the conference. Selected prize winners were:

- First place: Lauren Dillon, Northwestern University "Incorporation of Detailed Phase Behaviour and Secondary Reactions in Mechanistic Model of Fast Pyrolysis of Neat Cellulose";
- Second place: Joseph Polin, Iowa State University "Autothermal Operation of a Fluid-Bed Pyrolyser";
- Third place: Yawer Jafri, Luleå University of Technology "Entrained-Flow Co-Gasification of Black Liquor and Pyrolysis Oil – Concept Verification and Assessment of Gasifier Performance".

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Pacific Northwest
NATIONAL LABORATORY

IEA Bioenergy Task 34: The new Triennium

Doug Elliott of Pacific Northwest National Laboratory, US, and current Task 34 Leader discusses Task 34 in the new Triennium

The IEA Bioenergy Task 34 will continue in the new Triennium, which will run from 2016 to 2018. The title of the new Task will be Direct Thermochemical Liquefaction. 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 already committed to the Task are Germany, the Netherlands, Sweden, Finland, and New Zealand with leadership provided by the US.

Aims & Objectives

The overall objective of Task 34 will be to improve the rate of implementation and success of direct thermochemical liquefaction of biomass for fuels and chemicals by contributing to the resolution of critical technical areas and disseminating relevant information, particularly to industry and policy makers. The following are the Priority Topics identified for the Triennium by the Task:

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

Work Programme

The programme of work in the proposal for prolongation of the Task is based on the traditional approach of IEA Bioenergy with a well-defined technical and non-technical work programme in which small groups of participants will contribute.

wp1	Contribution to bio-oil standards development (CEN and ASTM);
wp2a	Round Robin for bio-oil analysis method validation (bio-oil REACH registration or CEN standard analytes);
wp2b	Webinar series on advanced methods of bio-oil/biocrude chemical analysis;
wp3	Stakeholder seminars;
wp4	Technoeconomic assessment;
wp5	Intertask collaboration-such as pretreatment of biomass for thermal conversion (with Task 32 and others) or Biofuel Trade (with Task 40 and others) including standardisation and transportation issues of bio-oil, and upgraded bio-oil;
wp6	Regular semi-annual meetings;
wp7	Publications.

Deliverables

The deliverables identified will include:

d1	Task 34 website maintenance;
d2	Newsletter, twice a year;
d3	Organisation and reporting of an international Round Robin on bio-oil/biocrude analysis;
d4	Topical report on advanced methods of bio-oil/biocrude analysis (outcome of webinars);
d5	Progress and annual reports, and a final Triennium report to the IEA Bioenergy ExCo.

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IEA Bioenergy Task 34: New Task Leader

Alan Zacher of Pacific Northwest National Laboratory, US, talks about his new role as Task 34 Leader

Welcome to the new Triennium for IEA Bioenergy Task 34, one that is characterised by many changes. We are broadening the Task to include both pyrolysis and hydrothermal liquefaction, we have a few changes in the member

(Continued on page 32)

IEA Bioenergy Task 34: New Task Leader ...continued

countries, and we have a number of new faces as we say farewell to some of our friends and colleagues.

I will be leading Task 34 this Triennium as well as becoming the US country leader. It would be arrogant to think that I adequately fill a chair that has been held by both Tony Bridgwater and Doug Elliott, both of whom have made legendary contributions to pyrolysis research over the years. Yet it is by standing on their shoulders that you and I can make even greater contributions; as together we will advance these various technologies further towards broad application.

"If I have seen further, it is by standing on the shoulders of giants." - Isaac Newton.

While I have collaborated with many of you, I would like to better introduce myself to the wider audience. I have worked for over 20 years at the Pacific Northwest National Laboratory (PNNL) in Richland, Washington. While receiving degrees in Chemical Engineering and Chemistry from Washington State University, I interned with the same PNNL research group that I later joined. I have worked on a variety of technologies with the same core research group for over 20 years.

My research focus has varied considerably throughout my career, but most of it is characterised by developing applied research solutions to environmental and energy concerns. This has included analysis, development, and deployment of novel technologies for treating hazardous wastes; support of the cleanup efforts at the Hanford nuclear site; development of various catalytic processes for producing chemicals from biomass and fermentation sources; giving practical training in managing threats in the global security environment; and continued development of processes towards fuels from renewable biomass sources and waste streams.

My experience in biomass-to-energy has been primarily in catalytic upgrading, but over the last few decades has also included pilot and bench scale work in production of fast pyrolysis bio-oils, hydrothermal liquefaction to bio-crudes, catalytic hydrothermal gasification for fuel gas production, and intermediate upgrading of bio-oils to refinery intermediates. I have worked almost exclusively on fast pyrolysis and upgrading since 2009, and eventually took over as programme manager of pyrolysis and upgrading at PNNL in 2012.

In working with applied research teams, I have earned 28 US patents and 30 foreign patents, all of them in biomass catalysis except for one. As a result, I was named a Distinguished Inventor of Battelle in 2008, and went on to become the PNNL Inventor of the Year for 2011. My greatest technology achievement is being a co-inventor of the Propylene Glycol from Renewable Sources (PGRS) process, an economically competitive route for producing propylene glycol (PG) from a non-petroleum source. This process is at the heart of a 100,000 metric tonne commercial scale facility built by Archer Daniels Midland producing industrial and USP grade PG. The PGRS process has won multiple awards, and for this I was a co-recipient of the American Chemical Society Green Chemistry Award in 2015.

I currently manage the primary programme at PNNL for development of the catalytic process for conversion of bio-oil to hydrocarbon fuel blend stocks and intermediates, as well as other industry focused research projects. Our current research focus areas are in quantifying the chemistry and properties of actual bio-oil intermediates during conversion, designing catalysts based on that understanding, improving process and catalyst lifetimes, and seeking ways to reduce risks and answer uncertainties that prevent commercial adoption of bio-oil upgrading.

I am looking forward to continuing the collaborative environment of Task 34 and the biomass liquefaction community, not only in formal collaborations, but also in helping to nurture personal relationships between research teams. These international partnerships, many of which have lasted for over 30 years, have produced steady progress towards our shared goals of advancing the adoption of renewable fuels in each of our own countries.

I believe that the challenges associated with the adoption of renewable biomass energy technologies are larger than the work of any one person, technology, organisation, 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.

This is why I am looking forward to working with you in the next Triennium.

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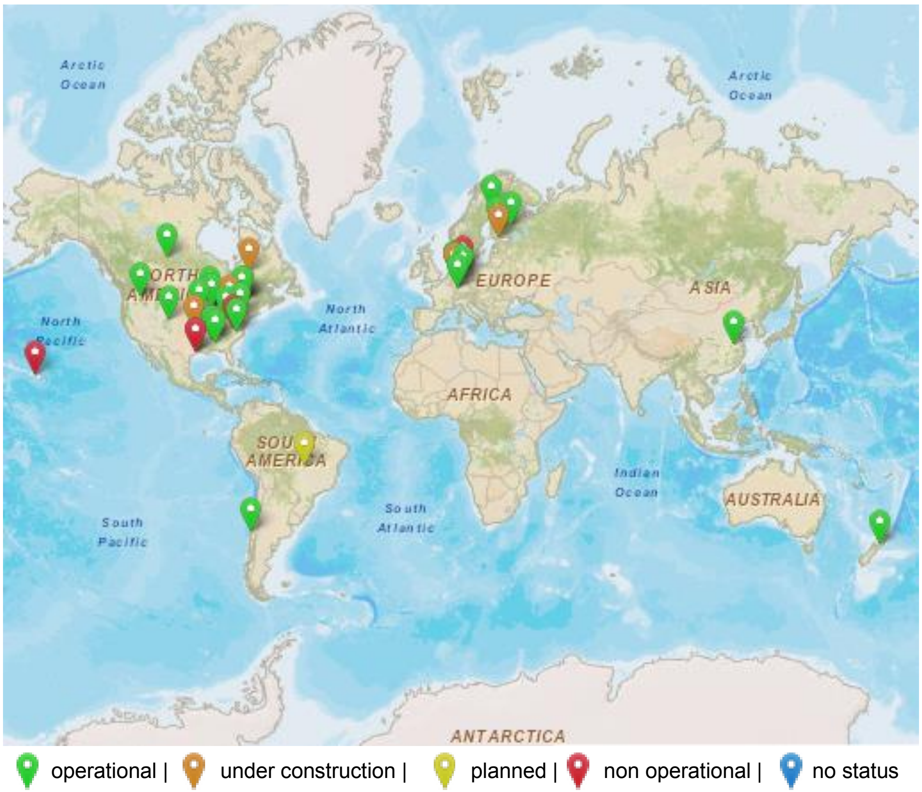
New resource: Pyrolysis Demoplants Database

The IEA Bioenergy Task 34 website - www.pyne.co.uk - has recently added a page dedicated to the new Pyrolysis Demoplants Database. This is a database of pyrolysis plants around the world with facilities categorised according to Technology Readiness Level (TRL), Technology and Status. Information is displayed both in list form and via an interactive map (reproduced in non-interactive form, *right*) enabling the user to quickly and easily find pyrolysis facilities across the globe.

Work on the database is ongoing and anyone aware of any plants not currently listed should contact Doug Elliott at dougc.elliott@pnnl.gov.

To access the database page, simply click on the link below:

www.pyne.co.uk



Pyrolysis equipment for sale

PyTEC Thermochemische Anlagen GmbH, Hamburg, Germany recently became insolvent. Their ablative pyrolysis system with a design capacity of 250kg/h is for sale. The complete system consists of a biomass bunker, rotating drum dryer, conveyer belt, feeding system to the pyrolyser (designed for wood chips, straw particles), quenching unit (spray tower, electrostatic precipitators), and oil tank. The whole system is computer controlled and operational. For further information contact Stefan Schöll (stefan.schoell@hamburg.de).

International Events

January 2016

13th-14th
The Future of Aromatics 2016
Amsterdam, the Netherlands

18th-19th
Fuels of the Future 2016
Berlin, Germany

20th-21st
Lignofuels 2016
Munich, Germany

February 2016

24th-26th
World Sustainable Energy Days
Wels, Austria

29th Feb-2nd March
ARPA-E Energy Innovation Summit
Washington DC, US

March 2016

3rd-4th
MBRE 2016
Glasgow, UK

6th-9th
Eco-Bio 2016
Rotterdam, the Netherlands

16th-18th
ibio 2016
Seoul, South Korea

23rd-24th
Gasification 2016
Rotterdam, the Netherlands

April 2016

3rd-6th
International Conference on Solid Waste Technology Management
Philadelphia, Pennsylvania, US

4th-6th
Green & Sustainable Chemistry Conference
Berlin, Germany

April 2016 (continued)

5th-6th
Wood Bioenergy Conference & Expo
Atlanta, Georgia, US

5th-7th
Argus Biomass 2016
London, UK

11th-14th
International Biomass Conference & Expo
Charlotte, North Carolina, US

May 2016

9th-12th
Pyro 2016
Nancy, France

23rd-26th
WasteEng2016
Albi, France

26th-28th
IRENEC 2016
Istanbul, Turkey

30th May-1st June
Renewable Resources and Biorefineries
Ghent, Belgium

June 2016

6th-9th
EUBCE 2016
Amsterdam, the Netherlands

14th-16th
20th Annual Green Chemistry & Engineering Conference
Portland, Oregon, US

19th-22nd
IConBM2016
Sicily, Italy

19th-24th
GPE 2016
Quebec, Canada

International events

FUELS OF THE FUTURE

The international conference “Fuels of the Future” is one of the most important forums for European players when it comes to discussing current issues in international biofuel policies, research and technological development. With a view to the changes to the European “Renewable energies” and “Fuel quality” Directives and to the report planned by the EU Commission on indirect changes in land utilisation from 2017, the congress represents an occasion for focusing on the further development and the consequences for the biofuel economy.

In January 2016, the mandatory greenhouse gas (GHG) emission reduction for fuels in Germany will have been applied in practice for one year: The mineral oil industry is thus legally obligated to reduce the CO_2 emissions of its fuels. Germany is the only EU member state in which this GHG quota applies exclusively. All other countries still have a biofuel quota which guarantees a specified sales volume.

A clear European fuel and mobility strategy extending beyond 2020 is needed to establish reliable framework conditions for investors.

PANEL 1
Basic conditions for future mobility with biofuels

PANEL 2
Podium discussion: biofuels in the transport sector—development until 2020 and beyond

PANEL 3
A One year of GHG reduction obligation in Germany: experiences gained (Room A6)
B Partner country France (Room A8)

PANEL 4
A Biodiesel (M1-M3)
B Blending of bioethanol in the EU and globally (M4-M5)
C Biomethane (M6-M7)
D Advanced biofuels (M8)

PANEL 5
A Biodiesel research (M1-M3)
B Bioethanol: research & development (M4-M5)
C Biomethane International (M6-M7)
D Biofuel trade (M8)
E Vegetable Oil (Room A8 exhibitors forum)

PANEL 6
Climate protection targets and implementation in the transport sector

13TH INTERNATIONAL CONFERENCE ON BIOFUELS
18-19.01.2016 CITYCUBE BERLIN
VISIT www.fuels-of-the-future.com

Pyro2016

Pyro2016 will offer a platform where scientists, from academia as well as industry, can meet to discuss the recent advances in pyrolysis science and technology.

Pyro2016 intends to provide a scientific programme with a good balance between fundamentals and applied work in various fields (biomass, organic geochemistry, wastes, polymers, art and forensics, etc.). Studies dealing with a multi-scale approach to bridge the gap between molecular mechanism and reactor development will be especially welcome.

Numerous attractive social events in Pyro 2016’s UNESCO World Heritage city will be proposed, including museum visits and a taste of French gastronomy…

www.pyro2016.com

International events

EUBCE 2016

AMSTERDAM - THE NETHERLANDS
6 - 9 JUNE 2016

24th European Biomass Conference & Exhibition

The EUBCE covers the entire value chain of biomass to conduct business, to network, and to present and discuss the latest developments and innovations.

The EUBCE is supported by European and international organizations such as the European Commission, UNESCO - United Nations Educational, Scientific and Cultural Organization, Natural Sciences Sector, EUBIA - the European Biomass Industry Association, WCRE - the World Council for Renewable Energy and further organisations.

The Technical Programme is coordinated by the European Commission, Joint Research Centre.

EUBCE's Mission & Vision

The EUBCE provides:

- A high-level Scientific Programme
- Parallel events

and it attracts:

- Participants from a wide-ranging background: researchers, engineers, technologists, standards organisations, policy and decision makers, financing institutions
- Research & industrial exhibitors

Detailed Conference Subjects

1. BIOMASS RESOURCES
2. BIOMASS CONVERSION TECHNOLOGIES FOR HEATING, COOLING AND ELECTRICITY
3. BIOMASS CONVERSION TECHNOLOGIES FOR LIQUID AND GASEOUS FUELS, CHEMICALS AND MATERIALS
4. BIOMASS POLICIES, MARKETS AND SUSTAINABILITY
5. BIOENERGY IN INTEGRATED ENERGY SYSTEMS

www.eubce.com

TCS 2016

Symposium on Thermal and Catalytic Sciences for Biofuels and Biobased Products

November 1 - 4, 2016
The Friday Center, University of North Carolina-Chapel Hill

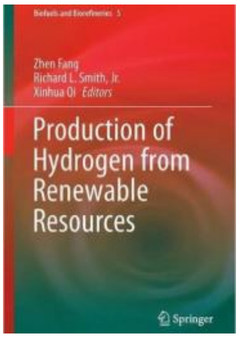
Save the Date!

Symposium on Thermal and Catalytic Sciences for Biofuels and Biobased Products

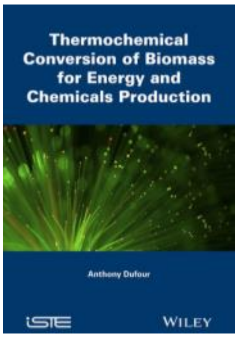
November 1 - 4, 2016
The Friday Centre, University of North Carolina-Chapel Hill

RTI International is coordinating this symposium.

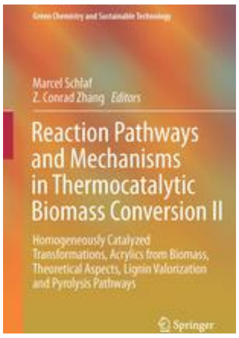
Publications



Production of Hydrogen from Renewable Resources
Published by Springer
Publication date: November 2015
Editors: Zhen Fang, Richard L Smith Jr, Xinhua Qi



Thermochemical Conversion of Biomass for Energy and Chemicals Production
Published by Wiley
Publication date: December 2015
Authors: Anthony Dufour



Reaction Pathways and Mechanisms in Thermocatalytic Biomass Conversion II
Published by Springer
Publication date: December 2015
Editors: Marcel Schlaf, Conrad Z Zhang

Computers & Chemical Engineering - Volume 82
Published by Elsevier
Publication date: 2 November 2015

Within this journal is the following paper:
A numerical study on biomass fast pyrolysis process: A comparison between full lumped modeling and hybrid modeling combined with CFD
Authors: Yu Ri Lee, Hang Seok Choi, Hoon Chae Park, Ji Eun Lee

BRISK Newsletter - Issue 8
Published by Aston University
Publication date: October 2015

PyNe 36

PyNe 37

Past editions of the PyNe newsletter are available on the website.

Further Information

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Birmingham, B4 7ET, UK

As the UK representative on the IEA Bioenergy Task 34, I would like to thank Kerri for all her hard work in collating and publishing fast pyrolysis news from around the world for the last two years. You can see for yourself how valuable her contributiuon has been and on behalf of all the PyNe readers, we wish her a happy and successful future.

- Tony Bridgwater

If you require further information about the PyNe newsletter, or you would like to contribute to future editions, please contact: e.wylde@aston.ac.uk.

www.pyne.co.uk

IEA Bioenergy

Task 34 - Pyrolysis

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