IEA Bioenergy

Task 34 - Pyrolysis

Welcome to Task 34

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By Doug Elliott, Task 34 Leader

The IEA Bioenergy Task 34 for Pyrolysis is well at work on its activities for the new triennium, which runs from 2013 to 2015. Current participants in the Task are Germany, the Netherlands, Sweden, Finland, and the UK, with leadership provided by the USA. This newsletter is produced by the Task to stimulate the interaction of researchers with commercial entities in the field of biomass pyrolysis.

We welcome our newest national team leader, from Sweden, Magnus Marklund.

Aims and objectives

The overall objective of Task 34 is 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 scope of the Task will be to monitor, review, and contribute to the resolution of issues that will permit more successful and more rapid implementation of pyrolysis 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:

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

In this issue of the newsletter

There are several articles from the

(Continued on page 2)

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Inside this issue

Welcome
...continued

participants describing the latest developments in fast pyrolysis including from the Netherlands, news on the production of aromatics by catalytic pyrolysis of biomass and plastic waste; from Germany, an overview of the R&D at Fraunhofer UMSICHT; from the USA we have contributions from USDA and NREL and a report from the tcbiomass2013 conference; from Finland a report from Fortum on CEN work and from the Finnish Petroleum Federation, a discussion of the need to standardise fast-pyrolysis bio-oil; and from the UK articles about bio-oil esterification and impacts of trace minerals. There is also an updated calendar of events of interest to the biomass pyrolysis community.

Please also check on the Task 34 website for updates to the participating Country Reports located elsewhere. These are short introductory articles from the national team leaders from each of the participating nations, 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.

You might also want to be aware of other updates to the Task 34 website, including to the "**Developments**" page and to the "**Task 34 Meetings**" section.

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District heating production with pyrolysis oil

A commercial application for expanding markets

Pyrolysis oil (renewable fuel oil (RFO)), among other potential solutions, enables the achievement of the national renewable energy target of 38% in Finland by 2020. This target, however, is just one of the challenges today's industry has to face. There are also concerns over indirect land use change (ILUC), the Renewable Energy Directive (RED), the Industrial Emissions Directive (IED), and the new Biomass Directive that is being drafted by the EU. These Directives have been, or are being, implemented on a national level.

A commercial application

Savon Voima Oyj, a local energy production and distribution company in the North Savo region of Finland, has built an RFO compatible district heating plant in Iisalmi. The plant can utilise both RFO and light fuel oil (LFO) as fuel, and its function is to produce hot water for the district heating grid. The hot water is used in heating up municipal and industrial buildings as well as private houses. Commercial use of pyrolysis oil will start at the beginning of 2014. (See related article by Finnish Petroleum Federation on page 10).

Figure 1: Commercial RFO compatible district heating plant.

Green Fuel Nordic Oy's biorefineries focus on meeting increasing market demand

The pyrolysis oil markets have now been opened in Finland, and Green Fuel Nordic (GFN) Oy is trying to meet the demand by building several biorefineries. The planning phase for the first GFN biorefinery in Iisalmi has been completed and construction work has started. Two other biorefineries are at the planning stage.

Green Fuel Nordic Oy's plan is to build up to 20 biorefineries in Finland, each producing 90,000 tons of renewable fuel oil (RFO) annually. As each stage of the new tight emission limits come into force the market demand can be expected to increase rapidly.

Green Fuel Nordic Oy is regularly sharing project status updates and other bioenergy related documents on its website www.greenfuelnordic.fi/media. One of the latest was a guideline for end-users on how to modify existing power plants to become bio-oil compatible.

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Figure 2: A 3D illustration of the GFN greenfield biorefinery in Iisalmi.

REACH registration of fast pyrolysis bio-oil

Pia Saari of Fortum Power and Heat Oy offers an update on the REACH registration process for fast pyrolysis bio-oil

In PyNe Issue 32 (December 2012), Sara Kärki of Fortum Power and Heat Oy in Finland wrote a comprehensive article on the REACH (Registration, Evaluation and Authorisation of Chemicals) registration of fast pyrolysis bio-oil (FPBO). The article described requirements for those companies which will manufacture or import fast pyrolysis bio-oil in the EU. The REACH registration process of FPBO took a leap during 2013 as the Joint Dossier was submitted to the European Chemicals Agency (ECHA) by the Lead Registrant in November.

The FPBO REACH consortium

This was established on 2 January 2013. By signing the consortium agreement and paying the entrance fee, all interested companies were able to join the consortium. Currently, the consortium consists of: BillerudKorsnäs AB, Envergent Technologies, Fortum Power and Heat Oy, Green Fuel Nordic Oy, Metso Power Oy, and UPM-Kymmene Corporation. Linnunmaa Oy was chosen to be the consortium manager by a common decision. The FPBO REACH consortium is small but functional and objective-oriented.

FPBO SIEF formation

Several companies had pre-registered their substance with the European Commission (EC) entry No. 302-678-6, "wood hydropyrolysed". This pre-SIEF (Substance Information Exchange Forum) consisted of both companies that intended to register fast pyrolysis bio-oil/liquid and companies that intended to register slow pyrolysis liquids. Pre-registrants were invited to a pre-SIEF meeting by Fortum in November 2012. During this formal meeting, there was a consensus that the fast and slow pyrolysis bio-oils are different substances according to the REACH substance identification provisions. Pre-registrants that were absent from the pre-SIEF meeting were provided with the opportunity to express their views afterwards. After the meeting, work to form a separate FPBO SIEF was started, and the final decision on the SIEF splitting was made in May 2013. At the same time, Fortum Power and Heat Oy was nominated as the Lead Registrant.

Substance identification profile

FPBO is a so-called UVCB substance (substance of Unknown or Variable composition, Complex reaction products or Biological materials). FPBO cannot be sufficiently identified by its chemical composition, because the number of constituents is large and the composition is, in a significant part, unknown and the variability of composition is relatively large. Therefore, the main identifiers of FPBO are the source and the process used.

In the tentative identification profile, the source was defined as "biomass, like wood". The consortium and SIEF members agreed on changing it into a more well-defined form, "lignocellulosic biomass". Thus, the following definition was agreed to be the final substance definition for FPBO:

“Liquid condensate recovered by thermal treatment of lignocellulosic biomass, at short hot vapour residence time (typically less than about 10 seconds) typically at between 450-600°C at near atmospheric pressure or below, in the absence of oxygen.”

The Chemical Abstracts Service (CAS)/International Union of Pure and Applied Chemistry (IUPAC)/ Public Name is Fast Pyrolysis Bio-oil, and the CAS number is 1207435-39-9. However, an EC number is not yet available because of the SIEF split from the pre-SIEF of “wood, hydropyrolysed” (EC number 302-678-6, CAS number 94114-43-9).

Those who consider their product to be similar to FPBO need to fulfill the presented definition. To support the decision on sameness, some additional properties and composition of FPBO have been defined. These are presented in Table 1. It should be kept in mind that these are parameters which have been set taking into account toxicological properties of FPBO, and not the typical quality characteristics.

Joint registration project

Those activities which were needed to jointly fulfill the registration obligation and other requirements laid down by the REACH regulation were, among others, performing the necessary laboratory tests, performing chemical safety assessments and compiling the chemical safety report, preparing the Joint Dossier,

(Continued on page 5)

REACH registration *...continued*

and managing the consortium.
An essential part of the registration project was the laboratory analyses. A sample, which was believed to present a typical FPBO process and to be composed of typical raw materials, was selected for testing. Laboratory testing for the sample was made based on the data gap analysis. The lab tests can roughly be divided into chemical analyses and physico-chemical, toxicological and ecotoxicological studies. Some of the ECHA-recommended tests could be avoided, because relevant data has been generated in the BIOTOX project (an assessment of bio-oil toxicity for safe handling and transportation, which was compiled in 2005).

The target was to submit the joint registration, covering annual volumes of more than 1000 tons, to the ECHA before summer 2013. However, there was a peak time in Good Laboratory Practice (GLP) laboratories due to the 31 May deadline for many other registrations, and therefore the testing for the FPBO sample was delayed within the planned schedule. The final joint dossier was submitted to the ECHA in November 2013.

Based on the test results and all relevant available research reports, a Chemical Safety Assessment, Chemical Safety Report, and Exposure Scenarios were produced. The Classification, Labelling and Packaging (CLP) classification was also updated. No study proposals were included in the joint dossier.

Table 1: Properties and composition of FPBO

pH	> 2 – 3.5
Water content	< 40 ww %
Ash content	< 0.5 ww %
Solids content	< 5.0 ww %
Viscosity (40°C)	< 200 mm^2/s
Density (kg/dm^3)	1.1 – 1.3 kg/dm^3
Polar components	
Formaldehyde	< 0.5 ww %
Methanol	< 3 ww %
Non-polar components	
PAH13 **a**	< 35 ppm
Bentso[a]Pyrene	< 0.01 ww %
Dibenz[a,h]anthracene	< 0.01 ww %
Sum of Carc. 1B classified substances **b**	< 0.1 ww %
Sum of Carc. 2 classified substances **c**	< 1.0 ww %

a Sum PAH13: Anthracene, Benz[a]anthracene, Benzo[a]pyrene, Benzo[a] fluoranthene, Benzo[k]fluoranthene, Benzoperylene, Chrysene, Dibenz[a,h] anthracene, Fluorene, Fluoranthene, Indenopyrene, Phenantrene, Pyrene
b Carc. 1B classified substances (Annex VI of CLP regulation 1272/2008): e.g. of sum PAH13: Benz[a]anthracene, Benzo[a]pyrene, Benzo[k] fluoranthene, Chrysene, Dibenz[a,h]anthracene
c Carc. 2 classified substances (Annex VI of CLP regulation 1272/2008): e.g. Formaldehyde, Acetaldehyde, Furfural

Letter of access

Companies planning to REACH register FPBO later on can join the joint registration via purchasing a Letter of Access (LoA) from the consortium. The LoA is a document which allows a SIEF member to refer to the entire Joint Submission, i.e. all studies used for the joint submission for a certain tonnage band. LoA is available per legal entity basis. The consortium is also willing to share data with other pyrolysis liquid registrants outside the scope of the substance identification via the read-across procedure. The price and more information on the LoA are available for any interested party upon request from the consortium manager (reach.fpbo@linnunmaa.fi) or Lead Registrant.

Next steps

Tasks of individual registrants during the following years include, for example, performing substance identity analyses according to the methods agreed by the consortium, and fulfilling the legal entity-specific REACH registrations requirements. Participation in consortium work and sharing relevant data with other consortium members will also be necessary. If the ECHA requests further information, this will need to be provided by the consortium.

Acknowledgement

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tcbiomass2013

Task 34 Leader Doug Elliott of Pacific Northwest National Laboratory reviews tcbiomass2013

‘tcbiomass2013’—the third 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. The conference was held in Chicago at the Sheraton Chicago Hotel and Towers on 3-6 September 2013, where the presence of participants from 19 countries and 145 organisations, including the Task 34 national team leaders, made for an energetic and engaging meeting. Attendees participated in three days of technological interchange with the world’s leading researchers, scientists and engineers in biomass conversion, with topics ranging from gasification and pyrolysis to upgrading and pretreatment.

tcbiomass2013 was organised by GTI under the leadership of Terry Marker and the scientific panel, with Vann Bush and Kyriakos Maniatis as conference co-chairs. Conference participants included senior experts and young researchers, major corporations and entrepreneurial startups, academics and plant operators, and the agenda featured five keynote speakers:

- Kevin Craig—US DOE perspective
- Robert Sturtz—Aviation Biofuel: Ready to Fly
- Ingvar Landälv—Thermochemical Pathway Developments in Europe
- Dr. Mitch Loescher—Path to Commercialisation of Drop-in Cellulosic Transportation Fuels
- Dr. Alan DelPaggio—Bridge Building 101

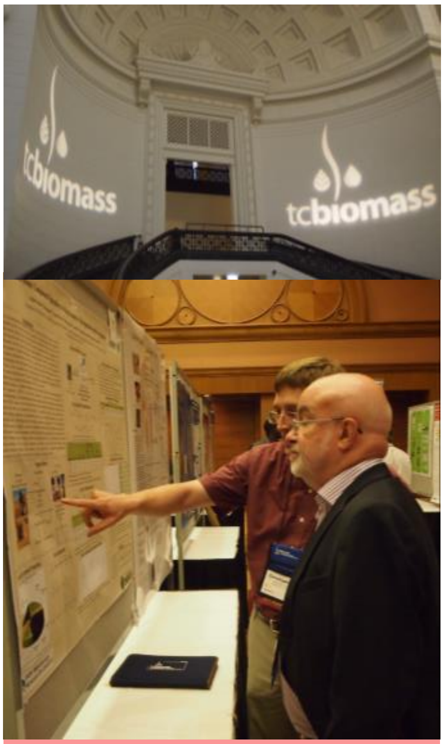
36 technical presentations were organised into sessions including:

- Gasification (2 sessions including 12 presentations)
- Pyrolysis (2 sessions including 12 presentations)
- Upgrading (9 presentations)
- Pretreatment and char (3 presentations)

The conference also included 130 poster presentations in the same topic areas. Continuing a trend in the recent thermochemical conversion conferences, the bulk of the presentations and posters related to pyrolysis, with 22 on gasification, 68 on pyrolysis, and 40 on upgrading, pretreatment and char. Presentations and many of the posters are available on the conference website.

tcbiomass2013 also hosted a competitive student poster contest to help connect today’s top engineering and science students with global bioenergy experts. Nearly 40 poster presentations were evaluated throughout the conference. Selected prize winners were:

- Kwang Ho Kim, from Iowa State University on “Hydrogen Donor Solvent Assisted Thermal Conversion of Lignin to Alkylphenols Using an Online Micro Reactor.”
- Paige Case, from the University of Maine on “Format-Assisted Pyrolysis of Biomass and Biomass Constituents.”
- Jan Ole Strüven, from University of Hamburg on “Catalytic Hydrocracking of Lignins in Sub-Critical Water to Aromatics products for Varied Feedstocks.”



The IEA Bioenergy Task 34 task presented a poster at tcbiomass2013.

The conference was reportedly a great success and one of the many highlights was the presentation of the Don Klass award, which was awarded to Dr. Esteban Chornet for Excellence in Thermochemical Conversion Science to recognise his innovative contributions in the field of bioenergy.

To view the presentations/posters from tcibiomass2013, visit: http://www.gastechnology.org/tcbiomass2013/Pages/2013-Presentations.aspx

tcbiomass2013
THE INTERNATIONAL CONFERENCE
ON THERMOCHEMICAL CONVERSION SCIENCE

Bio-oil stabilisation and upgrading by hot gas filtration

Robert Baldwin (above) and Calvin Feik (below) from the National Renewable Energy Laboratory (NREL) discuss the potential impact of hot gas filtration on the quality of bio-oil

The objective of this research project was to test the hypothesis that separation of char with its associated mineral matter from pyrolysis vapours before condensation would lead to improved bio-oil quality and stability. The primary unit operation that was investigated for this purpose was hot gas filtration. A custom-built heated candle filter system was fabricated by the Pall Filter Corporation and furnished to the National Renewable Energy Laboratory (NREL) for this test campaign. This system consisted of a candle filter element in a containment vessel surrounded by heating elements on the external surface of the vessel.

Alkali and alkaline earth metals, principally potassium, sodium, and calcium, are present in biomass. The concentration of total inorganics can range from 0.5 to 15wt% depending on feedstock. Alkali and alkaline earth metals in biomass are typically in the range 0.2 to 1% by weight. Alkali and alkaline earth metals have been shown to participate in or catalyse pyrolysis reactions leading to higher yields of char. In addition, it has long been suspected that alkali and alkaline earth metals may catalyse deleterious chemical reactions in bio-oil during storage, leading to increases in viscosity and giving rise to the well-known lack of stability for biomass pyrolysis oil.

Removal of char and associated minerals from pyrolysis oil for the production of biomass-derived boiler and turbine fuels has been demonstrated at NREL (previously known as the Solar Energy Research Institute (SERI)) using a ceramic cloth hot gas filter (HGF). Results showed that bio-oils with alkali and alkaline earth metal concentrations below 10 ppm could be produced, but stability of the oil was not demonstrated or quantified. The hot gas filter media used for these tests was a ceramic cloth fibre, which was found to 'blind' very quickly, leading to excessive back-pressure on the pyrolysis reactor; regeneration by periodic blowback was not successfully demonstrated and accordingly stable operation of the filter assembly was never reached during these early tests. Results of tests carried out on hot gas filtration at the University of Aston and the University of Twente have also been published with little improvement in the properties of the resulting bio-oil reported.

> **"The objective of this research project was to test the hypothesis that separation of char with its associated mineral matter from pyrolysis vapours before condensation would lead to improved bio-oil quality and stability."**

The metric used to evaluate stability in this case was a ten-fold reduction in the rate of increase of bio-oil viscosity. This metric is arbitrary, but was mandated by the US Department of Energy in the original Funding Opportunity Announcement (DE-PS36-08GO98018, Biomass Fast Pyrolysis Oil (Bio-oil) Stabilisation). Viscosity for all samples was measured following ASTM International's Standard D445. Accelerated aging of the bio-oil to simulate extended storage times was done by heating the bio-oil in closed containers for 8 hours and

(Continued on page 8)

Bio-oil stabilisation and upgrading ...continued

an additional 16 hours at 90°C with viscosity measurements made after each heating interval.

For these tests the pyrolysis reactor of NREL's 0.5 metric tonnes per day (MTD) process development unit (PDU) was operated in the entrained-flow mode at 500°C. The HGF test stand was installed on a slipstream from the PDU so that both hot gas filtered oil and bio-oil that was not hot gas filtered could be collected for purposes of comparison. Two filter elements supplied by Pall were tested in this project: 1) Grade H 310SC Porous Stainless Steel (PSS), and; 2) Dia-Schumalith™ 10-20 sintered silicon carbide ceramic. The hot gas filter was operated at 450°C for the PSS element and 420°C for the ceramic element in order to minimise loss of carbon efficiency associated with thermal cracking of the bio-oil. The test campaign on vapour-phase filtration of biomass-derived pyrolysis oil demonstrated that a bio-oil with substantially improved physicochemical properties can be obtained by application of hot gas filtration. The ceramic filter element and test stand supplied by Pall Filter Corp. and the vapour condensation and collection system designed and fabricated by NREL both demonstrated very good operability. Application of periodic blowback was shown to be effective in maintaining the filter element pressure drop within acceptable limits, and filter plugging was never experienced during the approximate 100 hour test campaign (over 1700 cycles).

> **"A bio-oil with greatly reduced alkali and alkaline earth metals and low solids content was produced by both filter elements."**

A bio-oil with greatly reduced alkali and alkaline earth metals and low solids content was produced by both filter elements. Bio-oil obtained by hot gas filtration with a porous sintered stainless steel (PSS) element had elevated iron content, suggesting that the material of construction is not suitable for this application. The PSS-filtered bio-oil also did not pass the viscosity metric of a ten-

(Continued on page 9)

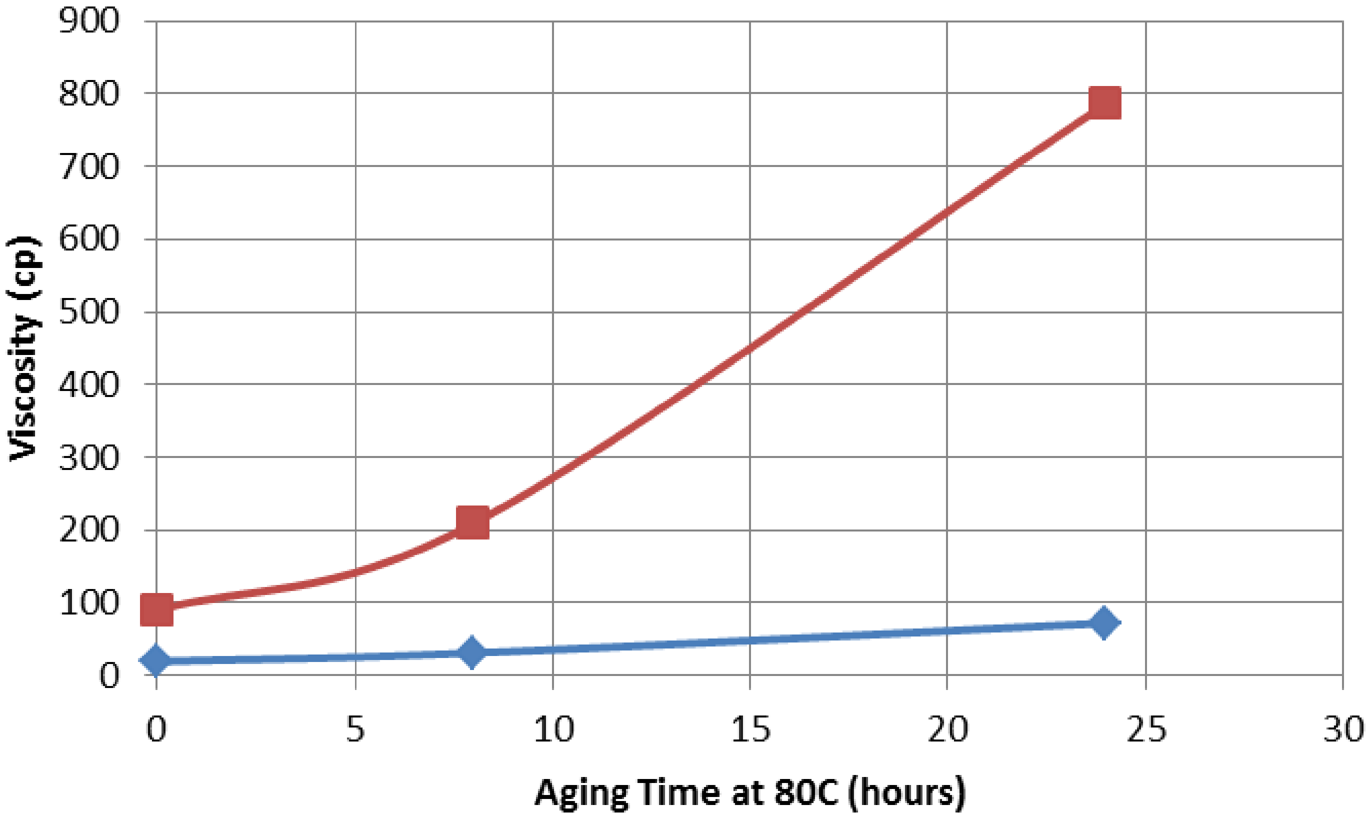


Figure 1: Accelerated aging test for raw and HGF oils (ceramic filter element); red line is the raw oil, blue line is the HGF oil; viscosity measurements made at 25°C.

Bio-oil stabilisation and upgrading *...continued*

Table 1: Properties of Hot Gas Filtered (Ceramic Filter Element) Bio-oil

	HGF Oil (420°C)
	wt %
C	41.12
H	7.87
N	0.08
O	50.92[a]
S	0.01
Ash	NDB[b]
VM	63.9
FC	6.4
Water	29.7
	ppm
Aluminum	<2
Calcium	<10
Chromium	<1
Iron	<5
Magnesium	<1
Manganese	<1
Nickel	<1
Phosphorus	<1
Potassium	<5
Silicon	<10
Sodium	<5
Zinc	<1

[a] including water

[b] ash not distinguishable from blank

fold reduction in the rate of viscosity increase as determined by the accelerated aging test at 80°C. Bio-oil obtained by hot gas filtration with a ceramic filter element was also low in alkali and alkaline earth metals, had nearly zero ash, and did not exhibit high iron content (Table 1). The ceramic-filtered oil passed the viscosity metric (Figure 1) indicating that this oil should be much improved with respect to storage and transport stability. Photographs of the fresh and used ceramic element are presented in Figure 2. In addition to low ash and alkali/alkaline earth metals, the hot gas filtered oil was found to have a molecular weight distribution that was significantly reduced in high molecular weight components when compared to non-filtered oil. The absence of inorganics and high molecular weight materials suggests that this oil should be less problematic in terms of fouling of catalysts in subsequent upgrading steps.

Figure 2: Fresh and used (~ 1740 cycles) ceramic filter element.

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NREL
NATIONAL RENEWABLE ENERGY LABORATORY

Initiation of standardisation of fast pyrolysis bio-oil under CEN

An explanation of the need for standards in the area of biofuel from Virpi Nummisalo of the Finnish Petroleum Federation

A new liquid fuel, biomass fast pyrolysis bio-oil, is coming to the market. Fast pyrolysis bio-oils can be a substitute for fuel oil or diesel in many stationary applications, including boilers, furnaces, engines and turbines for electricity generation. There are many projects to build up commercial-scale pyrolysis oil plants, and the first of these by Fortum Oy in Finland started operating in November 2013.

To be marketed commercially the product has to be standardised. Bio-oil is chemically different from conventional liquid fuels with somewhat different physical properties, and therefore must overcome both technical and marketing hurdles. To standardise bio-oil quality on the market, specifications are needed. Two burner fuel oil grades have been approved recently as ASTM (D7544) standards. Since the legislation and emission limits differ from Europe to North America, European (EN) standards are needed.

The use of pyrolysis oil as a fuel to replace heavy fuel oil in boilers and furnaces has been demonstrated. Fortum Power and Heat Oy will use its own bio-oil to replace heavy fuel oil in their heat plants. Fortum and a local Finnish energy company, Savon Voima Oyj, have agreed on starting a partnership in the delivery of bio-oil. The newly signed contract allows Savon Voima to replace heavy fuel oil in its heat production with Fortum's sustainable bio-oil. In the field of supplying bio-oil, Savon Voima is Fortum's first commercial partner in Finland.

The European Committee for Standardisation

The European Committee for Standardisation (CEN) has been given the mandate to develop each of the following:

- A European Standard for a quality specification for pyrolysis oil replacing heavy fuel oil in boilers.
- A European Standard for a quality specification for pyrolysis oil replacing light fuel oil in boilers.
- A Technical Specification for a quality specification for pyrolysis oil replacing fuel oils in stationary internal combustion engines.
- A Technical Specification for a quality specification for pyrolysis oil suitable for gasification feedstock for production of syngas and synthetic biofuels.
- A Technical Specification for a quality specification for pyrolysis oil suitable for mineral oil refinery co-processing.

Emphasis is given to the three first items mentioned above. The work will be undertaken in CEN/TC 19 in one so-called working group (WG 41 has been established). Pia Saari from Fortum Power and Heat has been chosen as a convenor and the Finnish Petroleum Federation acts as the secretariat. The kick-off meeting will be held in Finland at the beginning of this year.

The elaboration of standards should be undertaken in cooperation with the broadest possible range of interested groups, including international and European associations. Experts from outside Europe with experience of producing, using, transporting, and testing the product can become involved in standardisation. CEN has special rules for their participation. If you would like to participate in the standardisation effort as an expert, you should contact your own national standardisation body. An active participation is required.

For more information see:
http://www.fortum.com/en/mediaroom/pages/fortum-to-start-supplying-bio-oil-to-a-local-energy-company.aspx

http://www.cen.eu/CEN/Pages/faq.aspx

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FINNISH PETROLEUM FEDERATION

FarmBio^3 consortium: USDA-ARS and partners to demonstrate on-farm pyrolysis biorefining

Akwasi Boateng (above) and Charles Mullen (below) of the United States Department of Agriculture (USDA) discuss developments in the field of on-farm pyrolysis

The Agricultural Research Service (ARS), the principal intramural research arm of the United States Department of Agriculture (USDA) has been awarded a $6.86 million Biomass Research and Development Initiative (BRDI) grant from the National Institute of Food and Agriculture (NIFA) to lead a 14 member industry-university consortium to complete research and demonstrate distributed on-farm pyrolysis biorefining.

The project, "Distributed On-Farm Bioenergy, Biofuels and Biochemicals Development and Production via Integrated Catalytic Thermolysis" or "FarmBio^3" is being led by Akwasi (Kwesi) Boateng at the Eastern Regional Research Centre (ERRC, Wyndmoor, Pennsylvania, USA). The consortium is a regional network of partners dedicated to increasing the technology readiness level of biomass conversion to a level that will make on-farm bio-oil production feasible and sustainable. The project has four main areas of focus:

- Feedstock logistics;
- Pyrolysis reactor scale up;
- Catalyst development;
- Measurement metrices including techno-economic analysis and life cycle assessment with thermodynamic exergy assessment to advise on resource depletion.

The feedstock selected for FarmBio^3 encompasses a pool of three lignocellulosic biomasses important to US agriculture, including switchgrass, horse manure, and woody biomass from forest thinnings. The main goal of the feedstock portion of the project is to develop and optimise the production and logistics of existing biomass resources for use as feedstocks for local–and regional–scale production of high-quality pyrolysis oil. The consortium member institutions addressing feedstock tasks include Morrisville State College (New York), the University of Maine, and Mesa Reduction Engineering Inc. (New York), with input from farmers provided by the Montgomery County (Pennsylvania) Farm Bureau.

Feedstocks used included switchgrass, horse manure, and woody biomass from forest thinnings.

Scaling up from ERRC's 5 kg/h pyrolysis process development unit (PDU), "the Kwesinator", to a two metric ton per day (MTPD) farm demonstration unit is a major effort in this project. To do so, ARS will rely on its patent-pending design, which is based on a dual fluidised bed system dubbed the Combustion Reduction Integrated Pyrolysis System (CRIPS) and designed to mimic the fluid catalytic cracking (FCC) process.

A 10 kg/h proof-of-concept version of the CRIPS reactor has been successfully fabricated and started up in South Africa. Construction of the 2 MTPD unit at ARS has substantially progressed; this unit is being fabricated on a trailer so it will be mobilised to demonstrate and simulate distributed on-farm operation at various sites including those producing each of the focus feedstocks. Siemens is the industry partner on this part of the project and is designing controls for on-farm pyrolysis biorefineries. ARS expects the scaled mobile system to be ready for on-the-farm operation when more robust catalysts with longer operational life have been developed by the university and industry partners during this year.

(Continued on page 12)

FarmBio^3 consortium ***…continued***

FarmBio^3 has invested significant effort in catalyst development for both primary pyrolysis conversion (catalytic pyrolysis) and post production upgrading of bio-oils. For catalytic pyrolysis, the University of Oklahoma and Villanova University (Pennsylvania) are both working with ARS towards developing novel catalysts that are characterised by decreased production of coke, as well as increased reactivity, increased yield and carbon retention, while decreasing oxygen content of pyrolysates from the current state-of-the-art catalytic pyrolysis technology. Increased catalyst robustness and lifetime will allow feasible scaling up of this process in combination with the development of the CRIPS system. The team is working with PQ/ Zeolyst Corporation (Pennsylvania) for the eventual scale up of catalytic materials needed for on-farm operations.

To incorporate the deoxygenated intermediate into an existing petroleum refinery, several criteria will need to be met for infrastructure compatibility. Refinery catalysts are known to be subject to rapid poisoning and coking by trace amounts of impurities, thereby warranting the need for a prior hydrotreating step, and novel condensed-phase upgrading catalysts are being developed to achieve this. This task is accorded to a multi-university research team led by Professor J. Regalbuto's group at the University of South Carolina (USC). For this effort, the University of Delaware will begin with predictions of active catalyst metals using computational catalysis, robust and coke-resistant supports are being developed at Villanova onto which such metals will be deposited at USC for testing at the University of Maine using model reactions, and ultimately tested at ARS using real pyrolysis oil. A refinery partner, the American Refining Group of Bradford (Pennsylvania) is advising the consortium on criteria for petroleum refinery blending.

Figure 1: Members of the FarmBio^3 consortium pose in front of the 2 MTPD mobile pyrolysis unit which is under construction at ERRC in Wyndmoor, Pennsylvania.

On another track, the University of the Sciences in Philadelphia (Pennsylvania) is developing homogenous catalysts to break down pyrolytic lignin for precursor materials needed for the production of industrial chemicals.

Finally, the data from the future year on-farm demonstration of the above processes will be used to perform technoeconomic and life-cycle analyses, and to advise on the social sustainability aspects of distributed on-farm pyrolysis that will account for total resource depletion using exergy analysis. The task leader of this is effort is Drexel University (Pennsylvania) which is working in close collaboration with Swarthmore College (Pennsylvania), and SUNY -ESF (New York).

FarmBio^3 has just completed its first year. In August 2013, the entire team assembled for its first annual meeting at ERRC; an event that drew more than 50 participants including students, faculty, researchers, farmers, industry and business people from the member institutions and sponsors. Substantial progress towards the goals of the projects was reported by each of the consortium members. In the second year of the project, ARS is looking forward to further developments and the start up of the CRIPS demonstration unit.

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USDA United States Department of Agriculture

BioBTX aims for production of aromatics by catalytic pyrolysis of biomass and plastic waste

From top:
André Heeres, Niels J. Schenk, Erwin Wilbers, and Erik Heeres

An insight from André Heeres and Niels J. Schenk of BioBTX, and Erwin Wilbers and Erik Heeres of the University of Groningen

Aromatic compounds such as benzene, toluene, xylenes and naphthalenes are part of the primary petrochemicals playing an essential role in our society. Owing to environmental issues, a still growing energy demand, and the depletion of easily accessible oil resources, alternative routes for preparation have recently attracted significant interest. Furthermore, fluctuations in the bulk prices of aromatics (especially benzene and para-xylene), and the growing demand for green polyesters for the package and textile industry, have stimulated research within this field.

Whereas biomass can be utilised efficiently for industrial production of alcohols and alkenes, this is still not the case for aromatics. Routes currently under investigation for the preparation of aromatics from non-petrochemical sources are:

- Aqueous-phase reformation;
- Conversion of syngas into aromatics;
- Dehydration and aromatisation of pyrolysis oil and alcohols with zeolites;
- Production of p-xylene, a chemo-enzymatic route is taken involving the dimerisation/ dehydrocyclisation of renewable isobutanol.

Oxidation of “green” p-xylene to para-terephthalic acid (PTA) and subsequent polymerisation with green diols (ethylene glycol from bioethanol) provides a fully green route to polyesters currently used at large scale in various applications.

An ideal process yielding product mixtures fitting perfectly in the existing infrastructure of the petrochemical industry and avoiding multistep synthesis (“drop in concept”) is the catalytic pyrolysis of biomass and/or waste streams. It directly converts these materials to aromatics, usually using specific zeolite catalysts, and has been identified as the most promising route towards light aromatics derived from resources other than oil or coal. The yield and selectivity of the aromatics depends on the conditions used (temperature, ramping), the reactor, the nature of the biomass stream (lignin, cellulose, hemicellulose, wood, glucose, pyrolysis oil etc.), the catalyst used (structure, particle size, etc.) and, for example, the ratio biomass (waste)/catalyst.

Although progress has been made in the optimisation of aromatics from biomass and waste streams using catalytic pyrolysis, there is still a demand for a more efficient and commercially attractive process for preparation. BioBTX, located in the northern part of the Netherlands, aims to be the leading player in catalytic conversion of different biomass and waste streams in aromatics. In close collaboration with Professor Erik Heeres from the University of Groningen, a test installation was designed and supplied by BTG Biomass Technology Group capable of pyrolysing biomass catalytically into aromatics on a 100-500 g scale (Figure 1). Optimisation for different biomass and waste streams with respect to yield and selectivity of the individual aromatics is ongoing and should finally lead to a commercially attractive process fulfilling the market needs.

Besides a high yield of benzene, toluene and xylenes (BTX), the lifetime of the expensive

(Continued on page 14)

BioBTX aims for production of aromatics *…continued*

Figure 1: Bench scale catalytic pyrolysis unit for biomass and waste.

aromatisation catalyst is also an important factor for large scale production of aromatics from biomass/waste. During pyrolysis/ aromatisation the (zeolitic) catalyst is subject to extreme contamination with coke and inorganic material. Whereas the coke fraction can be burned off easily with oxygen, the catalyst gets severely contaminated with inorganic material originating from the biomass source during several cycles of regeneration. This will have an effect on the activity of the catalyst in the targeted conversion of hydrocarbons to aromatic compounds and, furthermore, it will shorten the lifetime of the catalyst significantly. BioBTX has solved this problem and has developed and patented an sophisticated two-step catalytic pyrolysis process for BTX production in order to reduce catalyst deactivation rates.

At the first stage, the biomass is catalytically pyrolysed to a vapour phase consisting of (unsaturated) hydrocarbons and oxygen containing compounds (furans, aldehydes, ketones etc). The vapour phase is subsequently aromatised in a second reactor with a zeolite catalyst. In a series of experiments, plywood sawdust with a particle size of 0.1 to 1.0cm was subjected to pyrolysis in a reactor based upon screw reactor technology. The catalytic pyrolysis was conducted with different catalysts; Catalyst 1 being, for example, a Broensted acid, and Catalyst 2 being a zeolite. Before use the catalysts were ground to a particle size of 160 to 650 micrometers and mixed with quartz sand in a weight ratio of catalyst to sand of 1:3. The catalysts were used in a catalyst to sawdust weight ratio (C/S) of 2.5–5.0.

The catalytic pyrolysis steps were conducted at different temperatures and different catalysts/feed ratios for a period of 0-12hrs at atmospheric pressure. The amounts of BTX were determined and calculated on the basis of the feed. Preliminary experiments showed that the catalysts remain equally active during several cycles and, surprisingly, in some experiments the total yield of aromatics for the two-step catalytic process exceeded the yields from a one-step aromatisation process (in identical conditions).

The ongoing current activities of the company have attracted a lot of attention from not only suppliers of different biomass and waste streams, but also large industrial players active in aromatics production and products derived thereof (e.g. terephtalic acid for green polyesters). In line with their business plan they expect to design and construct a larger demo -scale facility for the production of aromatics in the coming years. Furthermore, in the future, they will broaden their scope and expand their activities to other technologies attractive for the production of green aromatics and derivatives thereof (green polyesters). Ultimately, this should give a significant boost to the bio-based economy in the Netherlands.

Acknowledgement

Funding from the Innovative Action Programme Groningen (IAG3) is gratefully acknowledged.

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Pyrolysis research at the National Renewable Energy Laboratory

Kristiina Iisa, Peter N. Ciesielski (middle), and Mark R. Nimlos (bottom) give an insight into research progress at the National Renewable Energy Laboratory

The overwhelming majority of biomass pyrolysis research at the National Renewable Energy Laboratory (NREL) is supported by the **U.S. Department of Energy's Bioenergy Technologies Office**, and is focused on the production of "drop-in" transportation fuels. This includes studies of fast pyrolysis and vapour phase upgrading of pyrolysis vapours to produce hydrocarbon fuel blendstocks or refinery feedstocks. Experimental research is conducted from mg scale in laboratory reactors to kg scale in their pilot Process Development Unit, and their modelling work encompasses levels from molecular quantum-mechanics to Computational Fluid Dynamics (CFD) simulations.

Pyrolysis research

Research is underway to develop a better understanding of the chemistry and physics of biomass pyrolysis in order to improve our understanding of the composition of pyrolysis vapours and to help improve the yields of hydrocarbons during upgrading. Studies include the reaction mechanisms of the thermal decomposition of biomass and biomass pyrolysis vapours. Investigations of the thermal decomposition of a number of model compounds have been conducted using a hyperthermal nozzle [1] highlighting the reaction mechanisms and kinetics of decomposition reactions.

They are also investigating the impact of heat and mass transport restrictions on the formation of char and products. The microstructure of biomass is a complex, directional network of pores and cell walls that can vary substantially between different species of origin. We are developing methods to construct 3D models of heat and fluid transport of biomass particles with realistic geometry obtained from microscopy measurements of plant cell walls. These models are being used to understand the effects of microstructural parameters such as cell wall thickness and pore diameter on fast pyrolysis. The results from this work will enable reactor-scale models that accurately account for variations in biomass feedstock.

> **"They successfully demonstrated stable continuous filter operation with a biomass feed of 10 kg/h."**

Hot Gas Filtration

Hot gas filtration is another area of active interest for NREL. The impact of removing alkali and alkaline earth metals from pyrolysis vapours by hot gas filtration before condensation has been tested in the NREL Thermochemical Process Development Unit (TCPDU) in collaboration with Pall Filter Corporation, UOP, and other partners. They successfully demonstrated stable continuous filter operation with a biomass feed of 10kg/h: over 1700 filter cycles were completed with filter pressure drop remaining within design limits and with no plugging problems. They produced bio-oil with superior properties, including very low ash and alkali and alkaline earth metal contents, improved physico-chemical properties, and improved stability. This was demonstrated by a reduced rate of viscosity increase in the accelerated ageing test by more than a factor of ten. This work was recently featured in an article published in Energy and Fuel [2].

Vapour phase upgrading

Catalytic upgrading of biomass fast pyrolysis vapours is investigated in

(Continued on page 16)

Pyrolysis research at NREL *…continued*

order to produce pyrolysis oils with reduced oxygen content and improved physico-chemical properties. NREL's approach is to develop a better understanding of vapour phase upgrading to discover process conditions and new catalytic materials to improve yields of hydrocarbons.

Catalyst deactivation

Catalyst deactivation has a major impact on process efficiency and, using NREL's Molecular Beam Mass Spectrometer (MBMS), the gas phase products from pyrolysis and vapour phase upgrading can be monitored in real time to follow this process. Rates of deactivation and conversion mechanisms are investigated using this approach, as are process changes to reduce deactivation.

Hot gas filtration of pyrolysis vapours was shown to reduce alkali metal content in condensed oil, and it can also have beneficial effects on catalytic vapour phase upgrading. Filtration of the vapours dramatically reduces the build up of coke on catalysts, and NREL is investigating the addition of this process step to *ex situ* catalytic upgrading.

Compared to liquid transportation, fuels biomass is deficient in hydrogen, and the production of infrastructure-compatible drop-in transportation fuels at high yields requires hydrogen addition, either by hydroprocessing after pyrolysis, or the incorporation of hydrogen during the pyrolysis process. NREL has investigated light hydrocarbons as hydrogen donors for catalytic pyrolysis. Butane and propane in-laboratory and bench-scale experiments demonstrated reduced coking of the catalysts and suppressed oxygenate formation.

Technoeconomic modeling

NREL, in collaboration with PNNL, is working on techno-economic assessments of conceptual processes for liquid hydrocarbon fuels production via *in situ* and *ex situ* upgrading of vapours from the fast pyrolysis of biomass. As part of this effort, plausible future design cases will be elucidated showing the potential for cost competiveness of such processes with respect to petroleum-based fuels. High-level qualitative assessments for such processes were published in 2012 [3,4].

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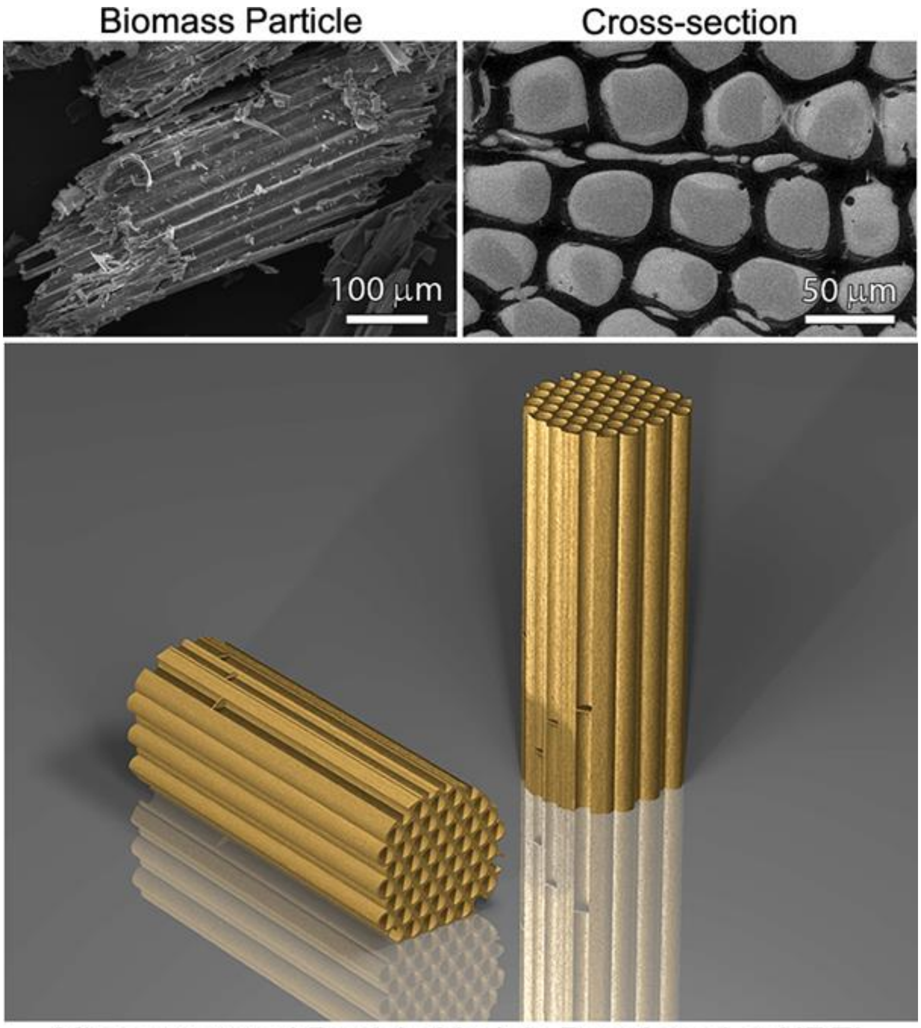

Figure 1: SEM images of biomass (top) that are used to develop three-dimensional models of biomass (bottom) for heat and mass transfer studies of pyrolysis.

(Continued on page 17)

Pyrolysis research at NREL *…continued*

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Pyrolysis research opportunities

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SEVENTH FRAMEWORK PROGRAMME

Hydropyrolysis of organosolv lignin

Jan-Ole Strüven from the Centre of Wood Science at Hamburg University, and Dietrich Meier of the Thünen Institute of Wood Research talk us through recent experiments on hydropyrolysis of organosolv lignin

Hydropyrolysis is a combination of pyrolytic degradation under hydrogenating conditions in the presence of catalytically activated hydrogen. Hence, radicals formed through homolytic cleavage of lignin bonds are instantaneously saturated and have little chance to repolymerize to form oligomers or coke. The process is typically performed at 350-400°C and 50-100 bar initial hydrogen pressure. This methodology is not new and has been described in literature extensively [1-10]. Process research activities in this field faded away in the early 1990s due to falling petroleum prices and less interest in bio-based platform chemicals. In conjunction with today's emerging biorefinery concepts, increasing oil prices, and climate change concerns, the use of hydrogen for upgrading biomass derived products is gaining interest again. To date, pressurized hydrogen is applied as a post-processing step for upgrading fast pyrolysis liquids (bio-oils) [11-13], and also as a fluidization and reaction medium within fast pyrolysis processes [14].

> **"In 2007 a German biorefinery project was initiated to investigate the fractionation of beech wood by an ethanol/water organsolv pulping process for further exploring the use of the resulting cellulosic and lignin fractions."**

In 2007 a German biorefinery project was initiated to investigate the fractionation of beech wood by an ethanol/water organsolv pulping process for further exploring the use of the resulting cellulosic and lignin fractions. Basically, two pathways were considered for lignin processing: conservation and modification of the polymeric structure for adhesives, films and foams, and degradation into phenols and other valuable aromatics by either alkaline cleavage or hydropyrolysis.

Hydropyrolysis experiments have been performed at Thünen Institute of Wood Research together with

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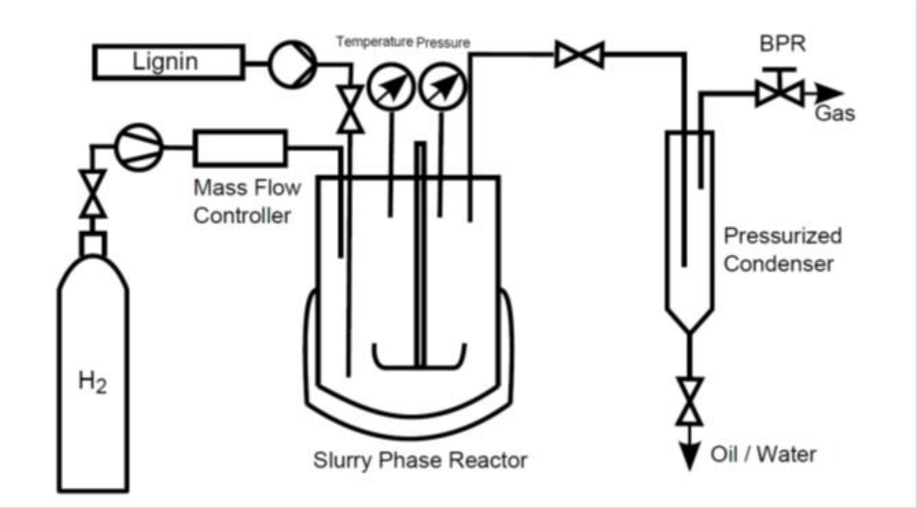

Figure1: Lab-scale reactor system for hydropyrolysis at the Thünen Institute of Wood Research.

Hydropyrolysis of organosolv lignin ...continued

partners from Hamburg University in a lab-scale reactor system (Figure 1). Slurries of lignin powder and water are prepared and filled either directly or via a high-pressure syringe pump into the stirred tank reactor. The reactor system can be operated in continuous, semi-continuous and batch mode. Liquid products are collected in a high-pressure condenser. By-product gas is vented and can be measured with a wet test meter for volume measurements. Samples can be collected in special gasbags for further gas chromatographic compositional analysis.

Numerous experiments have been executed under changing conditions; mainly catalysts and solvents were varied. For the sake of good mass and carbon balances the decision was made to use water as a slurry medium for the lignin powder. Maximum reaction temperatures were limited to 360°C in order to avoid gasification of lignin under supercritical water conditions.

A typical experiment runs as follows: 25g of lignin together with 3g Raney nickel and 50ml water are placed into a 250ml stirred autoclave. The reactor is purged with argon and hydrogen in order to get an oxygen free atmosphere. The autoclave is pressurized with hydrogen to either 25, 50 or 75 bars. After reaching the desired reaction time the pressure is released by opening a valve to a condenser in order to fractionate valuable monomers from the oligomeric residue.

Mass balance, GC/MS and SEC measurements have been conducted to ascertain the influence of initial H2 pressures and reaction times on the yields of phenols and dihydroxy-benzenes, as well as the molecular weight distribution of the obtained fractions.

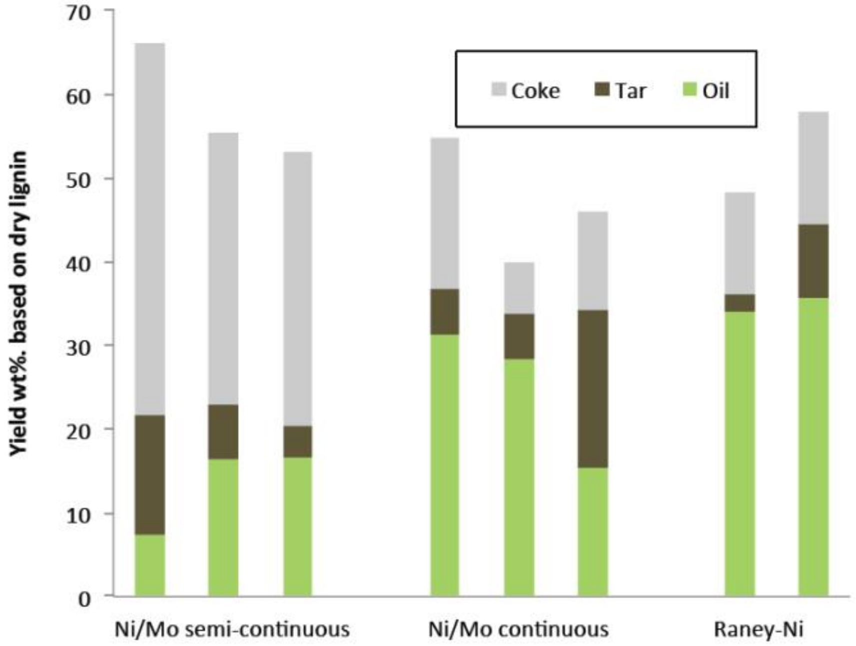

Figure 2: Influence of catalysts on liquid and solid product yields.

In the following diagrams some important findings are presented. Figure 2 compares the yields of liquid and solid products obtained with Ni/Mo and Raney-nickel catalysts. As the latter gave promising results, further experiments used exclusively various commercial Raney-nickel catalysts.

Liquid products were divided into dichloromethane solubles and acetone solubles. Figure 3 depicts molecular weight distribution

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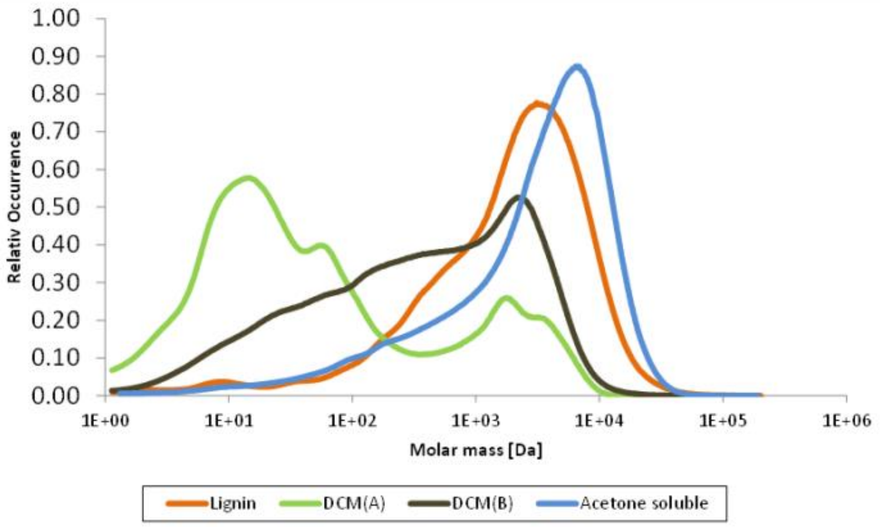

Figure 3: Molecular waste distribution curves of GPC measmurements from the starting material and the various oil fractions.

Hydropyrolysis of organosolv lignin *...continued*

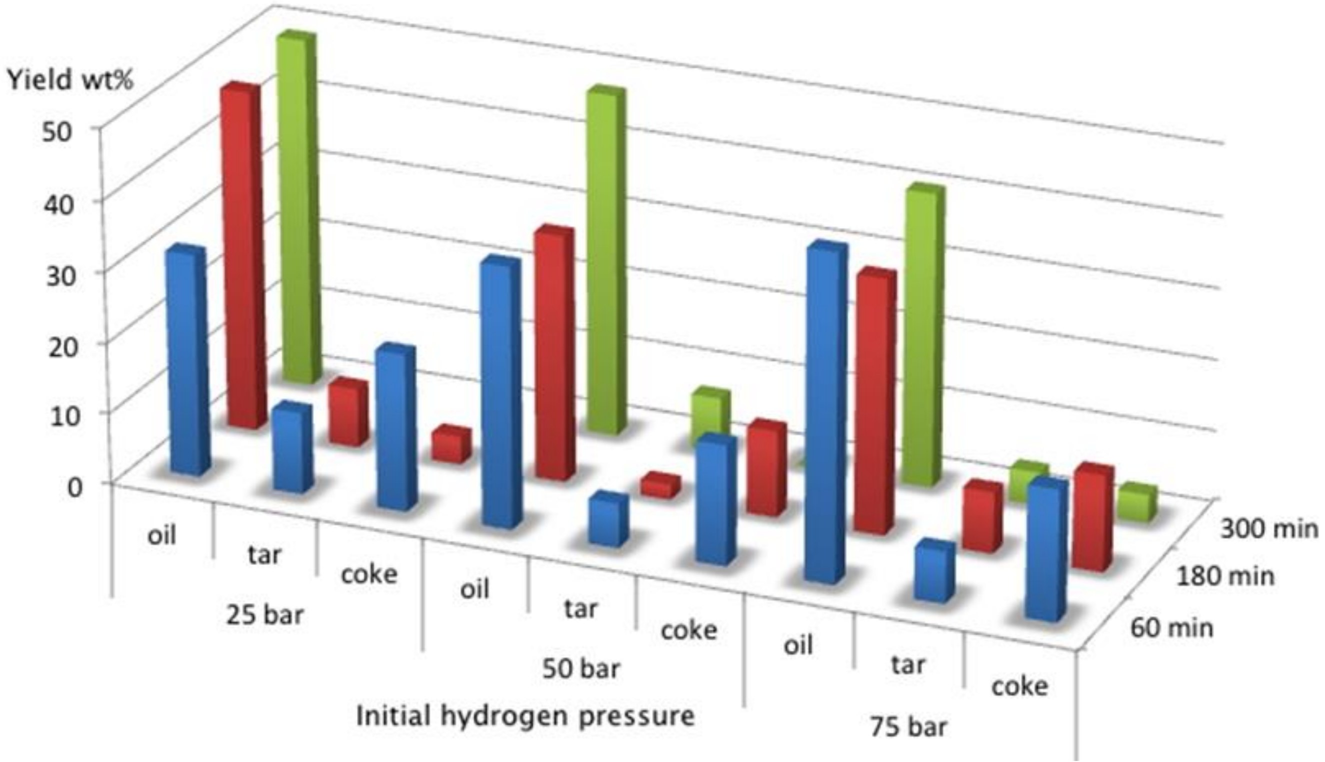

Figure 4: Oil, tar and coke yields from different operating conditions.

curves of GPC measurements from the starting material and the various oil fractions. The diagram shows both the depolymerization to lower molecular weight products (green & black lines) and some polymerization reactions (blue line).

Figure 4 summarizes yields on oils, tars, and coke from various experiments, indicating that highest oil yields are obtained at moderate initial hydrogen pressures and reaction times of 60 and 180 minutes. Coke is lowest at more drastic conditions.

Detailed gas chromatographic analyses revealed that the composition of monomeric aromatics depends on reaction time and hydrogen partial pressure as follows: longer times and lower hydrogen pressures give more phenols, whereas shorter times and higher pressures give more dihydroxybenzenes (catechols) as depicted in Figure 5.

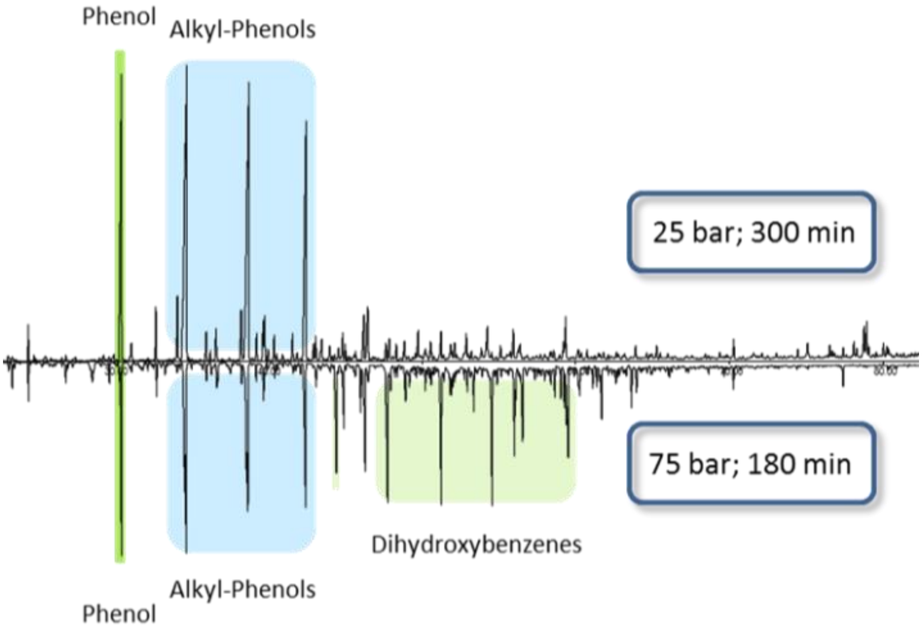

Figure 5: Gas chromatograms of oils obtained at different reaction conditions.

The following conclusions can be drawn from the present results:

- Online fractionation by releasing the pressure into a condenser delivers an oil which consists mainly of monomers and has a more valuable composition compared to pyrolysates

(Continued on page 21)

Hydropyrolysis of organosolv lignin *...continued*

obtained at 500°C by simple fast or slow pyrolysis.

- The yield of phenols increases with reaction time, but decreases with higher initial hydrogen pressure.
- The yield of dihydroxybenzenes seems to maximize within the first 60 minutes of reaction time, especially if a high initial hydrogen pressure is applied.

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Figure 6: Lab-scale reactor system for hydropyrolysis at Thünen Institute of Wood Research shown in Figure 1.

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Development of on-site flash pyrolysis units for stalk-like biomass

Tim Schulzke of the Fraunhofer Innovation Cluster, "Biomass", discusses the logistics of farm-based pyrolysis

> **"For experimental investigations Fraunhofer UMSICHT ordered two plants with different capacities: a laboratory plant from PYTRADE GmbH, Hamburg, and a pilot plant as design study from Claas Fertigungstechnik GmbH, Beelen (now MBB Fertigungstechnik GmbH)."**

The Fraunhofer innovation cluster, "Bioenergy", coordinated and operated by Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT, deals with the valorisation of different types of residual biomass from farming, as well as food/fodder production processes. The organisation and work of this cluster have already been published elsewhere [1].

Agricultural residues such as straw (from cereals, sun flower, canola, maize, etc.) remain over large areas after harvesting of the primary crop. If these residues are not left in the field and either burnt or ploughed into the earth, a recovery procedure is usually implemented. After grabbing, the straw is baled and then transported to a storage facility, either as a heap alongside the field, or in a barn. Barn storage is preferable for a later thermal conversion process as it offers better protection against the weather. From storage it is transported to the place of utilisation, e.g. a thermal conversion plant. Straw removed from agricultural fields contains several nutrients which must be replaced by mineral fertiliser in order to avoid impacting on subsequent crop yield.

The cost for the fertiliser replacement is the basis for the cost calculation for the recovered straw; if revenues for straw are below fertiliser price, it would be more economical to leave it in the field. Therefore the minimum selling price for loose straw in the field is in the range of €20/t. The additional costs of the logistics of moving and storing the straw total approximately €80/t, so that the minimum selling price of straw from storage is about €100/t. So, 20% of the cost originates from the nutrient value of the straw, and 80% originates from the logistics of transport and storage. This high share of the logistic cost in the selling price is the motivation for the development of a conversion process alongside the field or, even better, directly on the field, closely combined with grabbing.

There are many different pyrolysis technologies described in literature. From these publications it becomes clear that ablative flash pyrolysis might not be the best process for the pyrolytic conversion of straw into pyrolysis oil and biochar from an efficiency perspective, but it seems to be the only process with the potential of being operated on a moving platform. In ablative flash pyrolysis the biomass is pressed against a hot surface either by mechanical force or centrifugal force and the pyrolytic process occurs while the biomass is in contact with the surface. The efficient application of centrifugal force requires very small particles, whereas for the application of mechanical force larger geometries are preferable. Therefore, the decision for a system with mechanical force was reached, thus avoiding further grinding of the loose straw.

For experimental investigations Fraunhofer UMSICHT ordered two plants with different capacities: a laboratory plant from PYTRADE GmbH, Hamburg, and a pilot plant as design study from Claas Fertigungstechnik GmbH, Beelen (now MBB Fertigungstechnik GmbH). The laboratory plant for scientific investigations shown in Figure 1 has a flat plate as the hot surface. This is heated from behind with electrical resistance heaters and has a nominal capacity of around 15 kg/h of biomass feed, which is pressed against the plate with hydraulic cylinders. Investigations with different types of biomass (solid wood, wood shavings, several types of straw,

(Continued on page 23)

Development of on-site flash pyrolysis units for stalk-like biomass *...continued*

miscanthus, bagasse, etc.) can be performed. The loose biomasses must be briquetted to be used in this laboratory installation.

The pilot plant shown in Figure 2 represents the first step towards the future mobile application of the pyrolysis process for agricultural residues and has a nominal capacity of 100kg/h (expendable to approx. 1t/h, if the concept proves viable). The hot surface in this plant has a cylindrical shape and is heated from the inner surface with hot flue gases from a propane burner. Propane from bottles is used as fuel to provide the process with heat to have an open loop process for the first step. The combustible permanent gases remaining from the condensation process are measured in terms of volumetric flow rate and heating value to give an estimate of whether these gases contain enough energy to produce the process heat for the second step of development. The cylindrical shape was chosen because, after scale-up, a long cylinder fits better to road traffic licensing regulations than a large disk. The straw is pressed on the outer surface of the rotating cylinder by means of stuffing screws, which serve two purposes: compaction of the loose biomass into a continuous briquette, and application of the necessary mechanical force. Therefore, only stalk-like biomass as loose straw can be utilised in this pilot plant.

While the pilot plant is in the commissioning phase, the laboratory plant has already been used for benchmark pyrolysis tests performed with latches from beech and a mixture of spruce and fir. Samples of the pyrolysis oil were analysed at Thünen Institute for Wood Research in Hamburg, Germany, by Dietrich Meier and his team. For all surface temperatures investigated, no phase separation occurred. The oil has an average water content of 24.5%. The ultimate analysis gives a carbon content of 41%, a hydrogen content of 7.7% and an oxygen content of 51% (water content included). The lower heating value was around 15.7 MJ/kg. All of these values are well within the range of published data.

The laboratory pyrolysis plant was originally equipped for direct cooling of the pyrolysis vapours by means of a liquid quench. Parallel to the main cooling line, a so-called side slip was installed to collect liquid samples from the plant. Here, indirect cooling takes place via a coil cooler. To produce larger amounts of undiluted pyrolysis oil

(Continued on page 24)

Figure 1: Laboratory flash pyrolysis plant.

Development of on-site flash pyrolysis units for stalk-like biomass *...continued*

Figure 2: Design study for a “mobile” flash pyrolysis plant.

for further upgrading, the main condensation line was also equipped with a cooling coil for indirect heat exchange in the condensation of the pyrolysis vapours.

Several developments are planned for the future. The pilot plant will be put into operation and the laboratory plant will be modified to allow cylindrical briquettes as a fuel supply (to test straw pyrolysis). A staged condensation for a first separation of higher molecular hydrocarbons (sugars, phenolic oligomers, etc.), organic acids and alcohols (acetic acid, formic acid, methanol, etc.) and water is also being worked on in the side slip of the laboratory plant. Finally, in a joint research project with Thünen-Institute for Wood Research, funded by Fachagentur fuer nachwachsende Rohstoffe e.V. (FNR), investigations into pyrolysis oil upgrade processes will be made. The upgrading processes concerned are esterification of organic acid compounds with higher alcohols (reduction of acid number, removal of water), hydrotreating of raw pyrolysis oil and mild hydrotreating of esterificated pyrolysis oil.

The Fraunhofer innovation cluster, “Bioenergy”, is partly funded by the Ministry of Innovation, Science and Research of the state of North Rhine-Westphalia (MIWF) with funds from the European Fund for Regional Development (ERDF) and Fraunhofer Gesellschaft.

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Reference

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Spatiotemporal profiling of wood fast pyrolysis by STR-DRiSP

Paul Dauenhauer of the University of Massachusetts Amherst (above) and Jim Pfaendtner of the University of Washington Seattle (below) describe the STR-DRiSP technique

Pyrolysis of lignocellulosic biomass produces a small amount of solid char residue as well as gases and condensable vapours which can be collected and upgraded to biofuels and chemicals. The condensed vapours, referred to as 'bio-oil,' are comprised of a large number of highly oxygenated chemical species, some of which are difficult to catalytically upgrade. Despite the importance of bio-oil in producing renewable chemicals and fuels, the reactions and associated transport phenomena of fast pyrolysis remain difficult to describe and model [1].

Fast pyrolysis of lignocellulosic materials such as wood is particularly challenging to model due to the integration of complex biopolymer chemistry and combined heat/mass transport within microstructured wood particles. At high temperatures, wood fibres within pyrolysis reactors exhibit extreme temperature gradients, and reactions propagate across wood particles as an integrated thermal-reaction front consisting of multiple zones (Figure 1). Attempts to describe this process in the literature integrate lumped kinetic mechanisms of pyrolysis into one-dimensional (1D) reaction-transport models. However, the complexity arising from the large number of reaction-transport parameters convoluted with a rapidly changing lignocellulosic microstructure requires more detailed experimental data than currently is available via existing methods.

We reveal a new experimental technique, called 'Spatiotemporally Resolved – Diffuse Reflectance in situ Spectroscopy of Particles (STR-DRiSP)' [2], capable of characterising the carbohydrate composition of reacting lignocellulosic biomass particles with high spatial (10μm) and temporal (1ms) resolution. Via this technique, shown in Figure 1, incident light (for spectroscopic purposes) is applied to a 1x2x2 mm wood block of yellow poplar, and heat is applied to the bottom surface. As heat is applied sufficiently fast the reaction front takes on a 1D nature, and the surface of the particle mimics the internal composition.

Compositional characterisation of the reacting particle at the surface utilises a two-dimensional (2D) detector array with online spectral filtering, which allows for simultaneous focusing of independent detectors at different positions on the wood surface. The method takes advantage of the differences in diffuse reflectance of carbohydrates versus lignin/char in the range of 13,000-23,000cm-1, where lignin and char are highly absorptive. Calibration for the spectral response of carbohydrates and lignin/char allows for predictive composition based on detector response, leading to the experimental development of spatiotemporally-resolved compositional profiles of reacting wood particles as shown in Figure 2 (at 500°C).

The compositional profiles derived from STR-DRiSP were utilised in the development of a 2D reaction-transport model of reacting yellow poplar (not shown) [2]. In addition to compositional data the STR-DRiSP method measures particle shrinkage (visible in Figure 2), allowing for implementation within reaction-transport models of the fundamental relationships between particle composition, temperature, and cellular microstructure (liquefaction and collapse). Inclusion of prescribed particle shrinkage was found to be critical in achieving reasonable agreement with experimental data.

While STR-DRiSP compositional data and the associated reaction-transport model provide proof-of-concept, the technique opens the door for a future programme of

(Continued on page 26)

Spatiotemporal profiling of wood fast pyrolysis by STR-DRiSP *...continued*

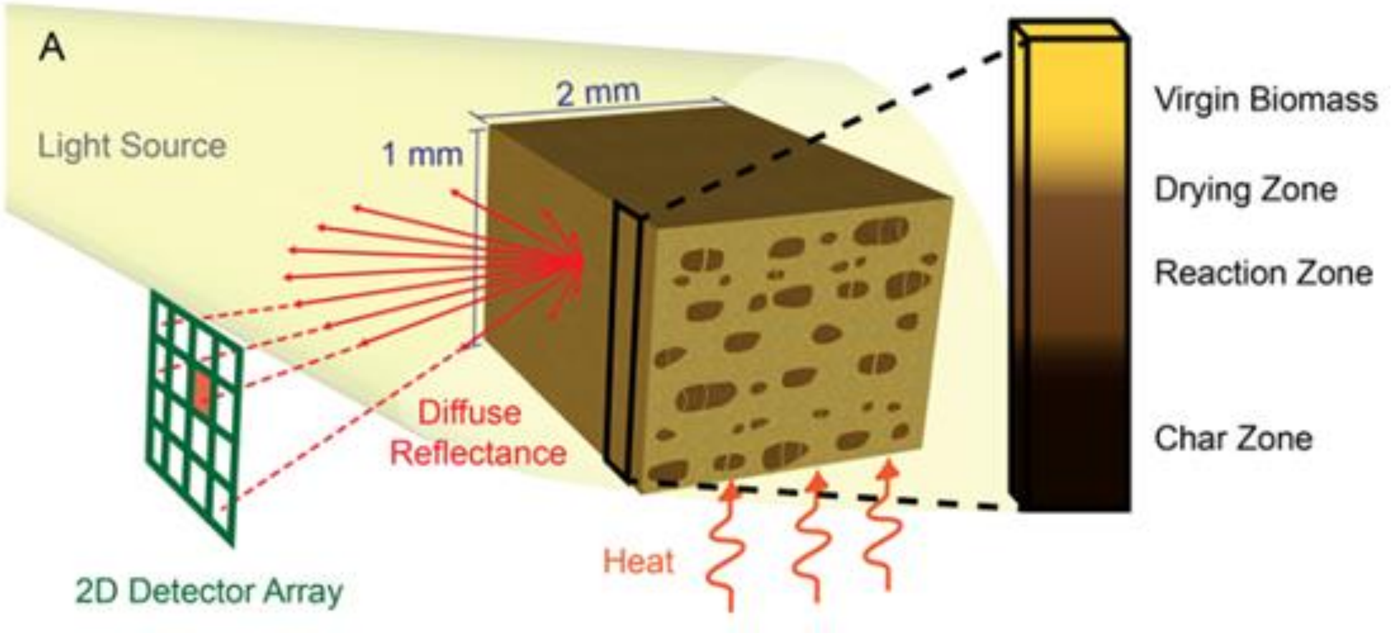

Figure 1: Characterisation of fast pyrolysis of wood fibres by Spatiotemporally Resolved - Diffuse Reflectance *in situ* Spectroscopy of Particles (STR-DRiSP).

research addressing many of the reaction and heat transfer phenomena associated with particle pyrolysis. Several longstanding research challenges remain, which will require the fine structure of STR-DRiSP compositional data, including:

- Poor understanding of integrated fast pyrolysis transport and reaction kinetics;
- Uncertainty about mechanisms of particle shrinkage;
- High variability in fast pyrolysis reaction-transport models [3].

Support

Work from the University of Massachusetts Amherst was supported as part of a subcontract with Cornell University, under Prime Agreement Award Number DTOS59-07-G-00052 from the United States Department of Transportation. Contributions from the University of Washington were supported by the National Institute for Food and Agriculture, U.S. Department of Agriculture, under Award Number 2011-68005-30416.

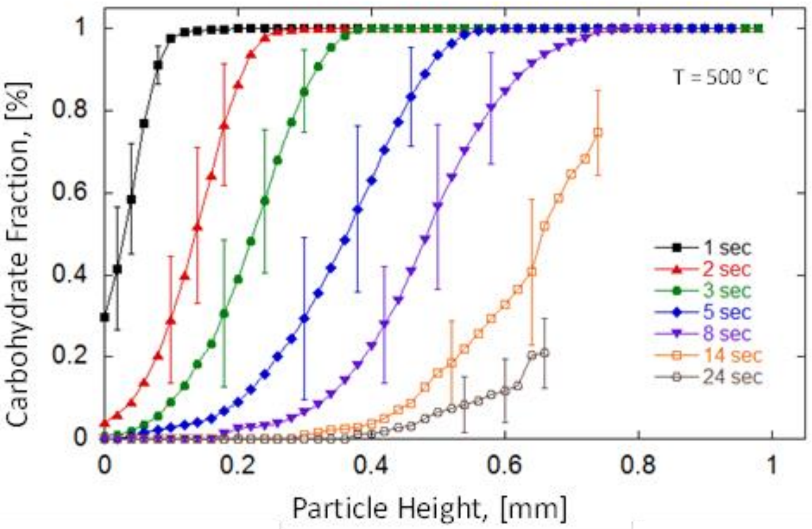

Figure 2. STR-DRiSP produces experimentally-derived spatiotemporal composition profiles of reacting particles of yellow poplar at 500°C.

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Effect of alkali metals on bio-oil from fast pyrolysis

Scott Banks of Aston University provides a summary of his PhD thesis

The liquid from fast pyrolysis of biomass, known as bio-oil, has some unusual characteristics that need to be addressed for wider use as an energy resource. Research at Aston University in the UK has investigated the influence of potassium and phosphorus on the physico-chemical properties of bio-oil.

Fast pyrolysis processing

A 1 kg h^{-1} continuous bubbling fluidised bed reactor was used to fast pyrolyse beech wood samples impregnated with potassium and phosphorus. The reaction unit is composed of three sections: the feeding system, the fast pyrolysis reactor, and product collection, and is shown in Figures 1 and 2. The reactor operated at 535°C with a hot vapour residence time of less than 1.5 seconds. Char was collected in two heated cyclones in series. Bio-oil was accumulated using a quench and electrostatic precipitator where the bio-oil was collected in a common collection tank from where it is usually run off at the end of an experiment.

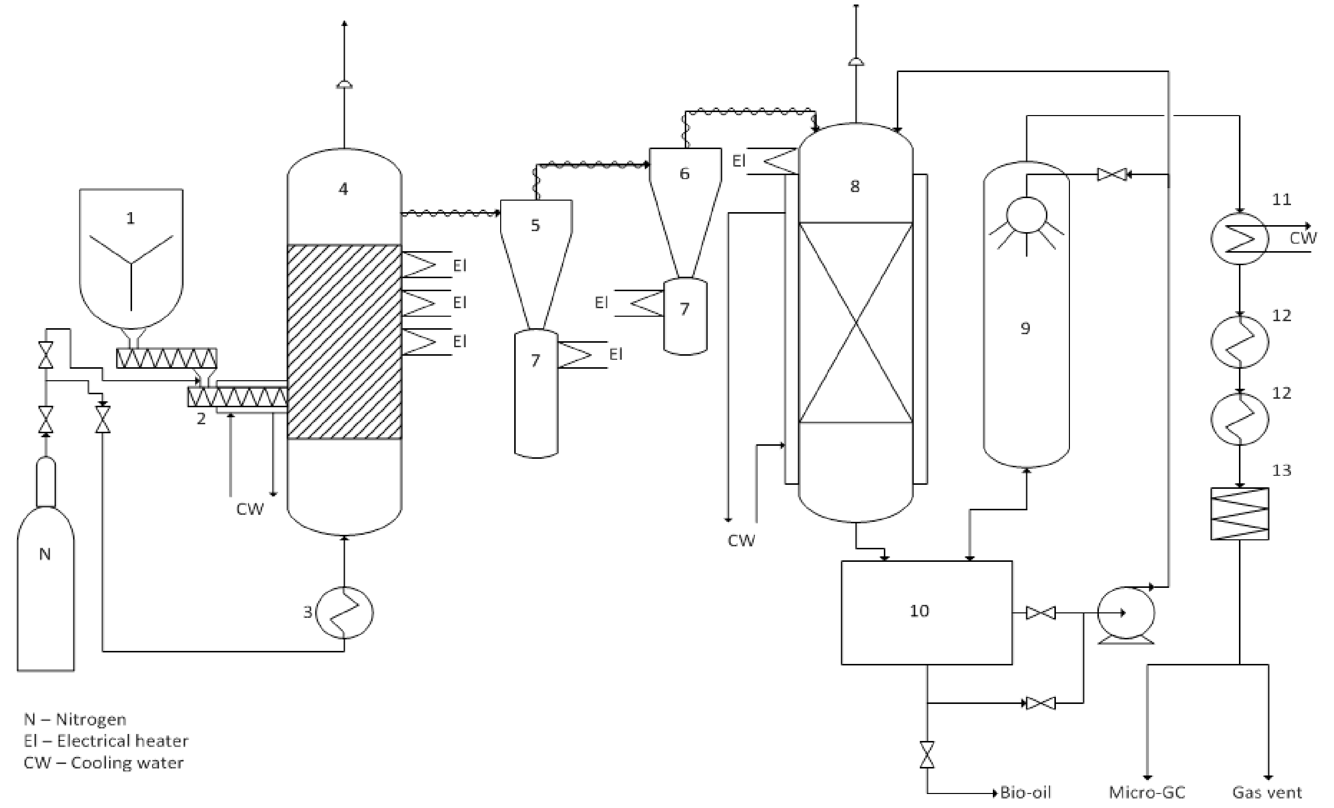
Feed modification

A wide range of experiments were carried out, but only the impact of potassium and phosphorus (key biomass ash components) is reported here. Standard beech wood samples were impregnated as follows:

- Impregnation with 1 wt.% potassium (as potassium acetate);
- Impregnation with 1 wt.% phosphorus (as phosphoric acid).

In addition a control of raw beech was also pyrolysed.

(Continued on page 28)



KEY: 1 – feed hopper, 2 – fast screw, 3 – nitrogen preheater, 4 – bubbling fluidised bed reactor, 5 – cyclone one, 6 – cyclone two, 7 – char pot, 8 – quench column, 9 – electrostatic precipitator, 10 – collection tank, 11 – water cooled condenser, 12 – dry ice / acetone condenser, 13 – cotton wool filter.

Figure 1: 1 kg h.-1 fast pyrolysis rig set-up.

Effect of alkali metals
...continued

Figure 2: Fast pyrolysis 1 kg h^{-1} rig.

Accelerated storage experiment
Accelerated storage experiments at 80°C for 24 hours simulate the effect of one year storage at ambient conditions [1]. Each sample has the dynamic viscosity measured by a Brookfield Viscometer model DV-II + pro rotational viscometer, and the water content was measured by Karl Fischer titration using a Mettler Toledo V20 KF titrator.

Each sample of bio-oil was then placed in an oven at 80°C for 24 hours, to simulate one year of storage at ambient temperature. After cooling, the viscosity and water contents were re-measured from which a stability index was calculated to determine the stability of the bio-oil. The stability index compares the change in viscosity and water to the original value. A completely stable bio-oil has a stability index of 1.00.

Results
The results are summarised in Table 1.

An increased potassium level resulted in higher reaction water from cracking of organics to water and carbon dioxide [2]. An increased phosphorus level resulted in an increased reaction water yield, indicating more dehydration reactions taking place during fast pyrolysis. The primary breakdown of the cellulose structure results in the main products being carbon dioxide, carbon monoxide, water and char. This dehydration of cellulose is favoured by low temperatures [3]. Char yields also increase due to acid catalysed condensation reactions [4]. Higher phosphorus content increases the char formation due to increased catalysis of condensation reactions.

Table 1: Values of viscosity and water stability indexes

Analysis	Beech wood	1.00 K	1.00 P
Stability index based on viscosity	1.29	2.24	1.93
Stability index based on water content	1.06	1.21	1.11

When the bio-oil samples were placed in the accelerated storage experiment an increase in viscosity for all impregnated samples was observed. The presence of char in bio-oil seems to catalyse reactions leading to the viscosity of the bio-oil increasing [5]. The water content index for K-impregnated beech wood bio-oil increases compared to raw beech wood bio-oil. Etherification and esterification occurring between hydroxyl, carbonyl and carboxyl group components results in water being a by-product [6]. Phosphorus impregnated beech wood bio-oil has a similar stability index based on water content to raw beech wood, suggesting that phosphorous has little or no effect on the water content of the bio-oil during storage.

Conclusions

- Both potassium and phosphorus catalysed fast pyrolysis increase char and reaction water yields.
- Potassium impregnated beech wood produced a phase separated bio-oil.
- Phosphorous impregnated beech wood produced a single phase bio-oil with an increased viscosity.
- An increase in viscosity for all impregnated samples was observed after accelerated storage.
- After accelerated storage, bio-oil produced from K-impregnated beech wood has a higher water content index compared to raw beech wood bio-oil.
- Bio-oil produced from P-impregnated beech wood has a similar water content index to raw beech wood bio-oil.

(Continued on page 29)

Effect of alkali metals *...continued*

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Figure 3: A – bio-oil obtained from raw beech wood (single phase), B – bio-oil obtained from 1% K impregnated beech wood (phase separated), C – bio-oil obtained from 1% P impregnated beech wood (single phase but higher viscosity).

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TCS 2014: Symposium on thermal and catalytic sciences for biofuels and biobased products

Join TCS 2014, a biofuels conference that focuses exclusively on thermochemical processing of biomass into fuels and chemicals. The programme will feature speakers discussing recent scientific advancements in this rapidly developing field. This forum offers a mix of plenary sessions featuring leading experts in the field, interactive breakout sessions, and ample opportunities to network with colleagues. The last day of the symposium will offer tours of the National Renewable Energy Laboratory. Programme topics include:

- Advances in pyrolysis and solvent liquefaction;
- Advances in gasification;
- Catalytic upgrading of bio-oil and syngas to fuels and chemicals.

Participants will include academic, government, and industry researchers who are making thermochemical processing one of the most exciting developments in advanced biofuels.

Location and hosts
The symposium will be held September 2–5, 2014, in Denver, Colorado, USA, at the Embassy Suites Denver Downtown Convention Centre.

The event is co-hosted by Iowa State University's Bioeconomy Institute and the National Renewable Energy Laboratory.

Dates for presenters (oral and posters)

- Abstract submissions open January 15, 2014
- Abstract deadline March 15, 2014
- Notified by May 1, 2014

For information on last year's conference, tcbiomass2013, see Task 34 leader Doug Elliott's review on page 9.

A "Call for Abstracts" will go out 15 January 2014. Visit http://www.tcs2014.org/ for details.

Pyro2014

20th International Symposium on Analytical & Applied Pyrolysis, Birmingham, UK, 19-23 May 2014

Marianne Bell of Aston University talks about Pyro2014

Continuing on from the success of the 19th International Pyrolysis Symposium, which was held in May 2012 and welcomed over 300 participants at Johannes Kepler University in Linz, Austria, we are now planning the 2014 conference which will be held in Birmingham, UK.

To book a place, submit an abstract, and for further information, please visit www.pyro2014.co.uk.

Sponsors and hardware vendors are welcome and will be offered an attractive conference package. For details please enquire with: m.bell3@aston.ac.uk.

Organised by Tony Bridgwater Director of the European Bioenergy Research Institute at Aston University, the conference will cover the latest results in all areas of pyrolysis and related thermal processes and will attract a wide range of researchers from academia and industry.

Topics will include:

- Analytical pyrolysis (including biomass, polymers, etc.);
- Applied pyrolysis (including biomass, polymers, waste, bio-oils, biochar, etc.);
- Catalysis in pyrolysis;
- Pyrolysis kinetics and mechanisms;
- Reactive pyrolysis.

Speakers at Pyro 2012, held at Johannes Kepler University in Linz, Austria.

www.pyro2014.co.uk

Table 1: Prices and Discounts for Pyro2014.

	Early Bird	After 1 April 2014
Regular	£550	£650
Student	£425	£525
Accompanying Person	£140	£160

Table 1: Important Dates for Pyro2014.

Abstract submission deadline	01 February 2014
Early bird registration deadline	31 March 2014

International Events

FEBRUARY 2014

13th
Westminster Energy, Environment and Transport Forum: Biofuels in the UK: targets and land use, advanced biofuels and next steps for policy
London, UK

26th-27th
Energy from Waste 2014
London, UK

26th-28th
World Sustainable Energy Days 2014
Wels, Austria

MARCH 2014

11th-13th
International Bioenergy Conference 2014
Manchester, UK

18th-19th
Bioenergy Fuels and Products Conference and Expo
Atlanta, USA

18th-20th
Green Polymer Chemistry 2014
Cologne, Germany

24th-26th
International Biomass Conference and Expo
Orlando, USA

APRIL 2014

1st-2nd
European Biomass to Power
Copenhagen, Denmark

1st-3rd
Sustainability Live 2014
Birmingham, UK

7th-11th
4th International Congress on Green Process Engineering
Seville, Spain

23rd-25th
Bioenergy and Biomass Utilisation Summit (BBS 2014)
Shanghai, China

APRIL 2014 ...continued

28th-29th
Euroheat and Power 2014
Brussels, Belgium

MAY 2014

4th-7th
iconBM International Congress on BIOMASS
Florence, Italy

12th-14th
5th AEBIOM Bioenergy Conference
Brussels, Belgium

12th-14th
11th BIO World Congress on Industrial Biotechnology
Philadelphia, USA

19th-22nd
6th International Freiberg Conference on IGCC and XtL Technologies (IFC 2014)
Dresden Radebeul, Germany

19th-23rd
Pyro2014—20th International Symposium on Analytical and Applied Pyrolysis
Birmingham, UK

29th-24th
13th International Symposium on Bioplastics, Biocomposites and Biorefining (ISBBB 2014)
Guelph, Canada

JUNE 2014

3rd-5th
World Bioenergy 2014
Jönköping, Sweden

4th-6th
10th International Conference on Renewable Resources and Biorefineries
Valladolid, Spain

23rd-26th
European Biomass Conference and Exhibition (EU BC&E)
Hamburg, Germany

Events

International Bioenergy Conference 2014

Research Councils UK Energy For a Low Carbon Future

To register for the conference please visit: www.bioenergy2014.co.uk

'SHOWCASING EXCELLENCE IN SUSTAINABLE BIOENERGY RESEARCH AND DEVELOPMENT'

Manchester Central Convention Complex, Manchester, UK, 11th-13th March 2014

Supported by the UK Research Councils and the Technology Strategy Board

This **International Conference will showcase UK bioenergy research in a global context** and will bring together **world-class researchers in bioenergy**, including academia, policy makers, industrialists and other related stakeholders. It will include **high profile plenary keynote speakers, subject-specific break-out sessions, workshops and poster presentations** that will cover:

- Biomass feedstocks for renewable energy generation;
- Biochemical and thermochemical conversion technologies, including biorefining;
- Sustainability: environmental, social, economic;
- Whole-systems modelling and analysis;
- Added value through alternative applications for biomass feedstocks;
- Industrial implementation and scale-up;
- Policy implications and needs.

The Largest Biomass Conference in North America

INTERNATIONAL BIOMASS CONFERENCE & EXPO

a BBI International event

ALL THINGS BIOMASS

PELLET SUPPLY CHAIN SUMMIT CO-LOCATED MARCH 24, 2014 ORLANDO, FLORIDA LEARN MORE

March 24-26, 2014
Orange County Convention Center
Orlando, FL

The **2014 International Biomass Conference & Expo** programme will include 30-plus panels and more than 100 speakers, including 90 technical presentations on topics ranging from anaerobic digestion and gasification to pyrolysis and combined heat and power, all within the structured framework of four customised tracks:

- Pellets and densified biomass
- Biomass power and thermal
- Biogas and landfill gas
- Advanced biofuels and biobased chemicals

At the 2013 event in Minneapolis . . .

100% of the exhibitors positively rated the quality of the entire conference

96% of exhibitors made valuable contacts

94% of exhibitors would recommend this conference

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http://www.biomassconference.com

Events

EU BC&E 2014

22nd European Biomass Conference and Exhibition

CCH - Congress Center Hamburg, Germany • 23 - 26 June 2014

The EU BC&E is the event in which the members of the bioenergy community can get a broad picture of the situation and trends emerging in today's market. The number of participants at the 2013 edition anticipate the size of the event in 2014 in Germany. It is set to be the European biomass highlight of the year, dedicated to sustainable energy with a high international acclaim.

The Conference provides a high-level scientific programme and parallel events which attract participants from a wide-ranging background: researchers, engineers, technologists, standards organisations, financial institutions, policy makers and decision makers.

http://www.conference-biomass.com

This event is supported by European and international organisations. The Technical Programme is coordinated by DG Joint Research Centre of the European Commission.

Early bird deadline
23 April 2014

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http://www.conference-biomass.com/Contact.111.0.html

WasteEng14

5th International Conference on Engineering for Waste and Biomass Valorisation and 1st WasteEng Summer School

Rio de Janeiro (Brazil)
August 25-28, 2014

(August 28-29, 2014)

Organised by

WasteEng Conference Series
WTERT Brasil
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Co-organised by

Earth Engineering Center
Princeton University
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WasteEng2014 Conference (August 25-28, 2014)

After four very successful previous issues WasteEng2005 (Albi, France), WasteEng2008 (Patras, Greece), WasteEng2010 (Beijing, China) and WasteEng2012 (Porto, Portugal) we are pleased to invite you to participate in **WasteEng 2014: 5th International Conference on Engineering for Waste and Biomass Valorisation to be held in Rio de Janeiro, Brazil.**

1st WasteEng Conference Series Summer School (August 28-29, 2014)

The 1st WasteEng Conference Series summer school will offer exclusive programmes designed in close collaboration with leading experts. This will give you a unique opportunity to develop your engineering skills in an industrial setting and work with highly topical technological cases. You will get the opportunity to work in teams with students and young engineers from many different countries and with many different engineering profiles. The courses will be taught by some of the best profiles within the area of renewable energy, and you will be introduced to a new approach to problem solving. The American Institute of Chemical Engineers (AIChE) will grant the courses with credit such as CEU (Continuing Education Unit) and PDH (Professional Development Hour).

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On-line registration, submission, payment:
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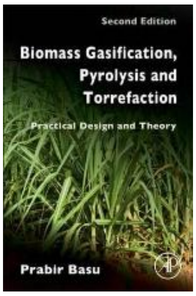
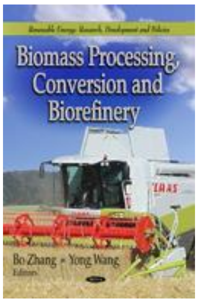
http://www.wasteeng2014.org/

Publications



Biomass Gasification, Pyrolysis and Torrefaction: Practical Design and Theory (Second Revised Edition)
Published by Academic Press (Elsevier).
Publication date: September 2013.
Author: Prabir Basu.



Biomass Processing, Conversion and Biorefinery
Published by Nova Science Publishers Inc.
Publication date: July 2013.
Edited by: Bo Zhang and Yong Wang.

Sustainability and Process Development: Developments in Sustainable Chemical and Bioprocess Technology
Published by Springer.
Publication date: December 2013.
Edited by: Pogaku Ravindra, Awang Bono, and Christopher Chu.

EERA Bioenergy NEWS—Issue 2
Published by Aston University.
Publication date: October 2013.

BRISK Issue 4
Published by Aston University.
Publication date: December 2013.

To visit the website of each of these publications click on the relevant title to open the hyperlink.

Past editions of PyNe newsletters are available on the website.

PyNe Issue 32

IEA Bioenergy
Task 34 Pyrolysis
PyNe 32
December 2012
Welcome to Task 34
By Doug Elliott, Task 34 Leader

IEA Bioenergy
Task 34 - Pyrolysis
Welcome to Task 34
PyNe Issue 33
June 2013
By Doug Elliott, Task 34 Leader

PyNe Issue 33

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IEA Bioenergy

Task 34 - Pyrolysis

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