

Sweden's representative in Task 34

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By Magnus Marklund of ETC in Piteå, Sweden

IEA Bioenergy Task 34 is pleased to introduce the country leader for our newest member, Sweden

The Swedish Energy Agency intends, through its participation in IEA Bioenergy, to work to strengthen Swedish knowledge and interests in the international arena and to enable Swedish actors to receive international knowledge about bioenergy via networking. The Swedish National Team Leader of IEA Bioenergy Task 34, Magnus Marklund, is now leading the international monitoring of general Swedish interests and opportunities for technology development and commercialisation of fast pyrolysis of biomass. Magnus is currently the Managing Director at ETC in Piteå, Sweden, and his main background is in gasification with the more recent addition of pyrolysis through



Figure 1: Magnus Marklund, Managing Director at ETC, studies a fractionated oil sample obtained from the ablative pyrolysis oil cyclone and staged condensation system at ETC.

the pyrolysis activities performed at ETC.

"The main overall aim of Sweden's participation is to push for an expansion of the use of the pyrolysis route in Sweden and to develop international cooperation within the Task. Pilot-scale experimental R&D work on fast pyrolysis in Sweden is currently being performed at the Royal Institute of Technology (KTH), (Continued on page 2)



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dealing with catalytic steam FB pyrolysis, and at ETC around ablative cyclone pyrolysis," says Magnus.

The current focus of experimental work at KTH is on confronting the challenges of reducing the internal oxygen present in the pyrolysis oil to levels suitable for the existing petrochemical industry, and at ETC to understand the underlying phenomena and observable facts related to condensation and cooling of pyrolysis oil.

Furthermore, studies on how tailor

made zeolite catalysts function in upgrading pyrolysis oil to highgrade hydrocarbon compounds are being performed at Luleå University of Technology together with ETC. More around the latter will be presented in upcoming issues of PyNe and more on the KTH work on their catalytic steam pyrolysis process can be found in a separate article by Efthymios Kantarelis in the current issue.

"Since BillerudKorsnäs decided to discontinue their NER300 pre-study on production of green oil at the end of last year, we are in need of new initiatives on pyrolysis activities in Sweden in order to regain momentum for the possible implementation of fast pyrolysis in the Swedish industry. Ongoing discussions between researchers, industry and public agencies will hopefully result in fruitful joint activities by the end of this year," Magnus concluded.



Members of IEA Bioenergy Task 34: 2013-2015



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Review of developments of fast pyrolysis in mobile processing systems



Doug Elliott of Pacific Northwest National Laboratory (PNNL) in the USA discusses the development of mobile pyrolysis units

The more common vision for fast pyrolysis of biomass for the production of liquids involves moderate-scale, site-based processing systems. However, there remains considerable interest in the development of small-scale systems for mobile processing of isolated biomass resources. Much of this work involves some variation on an auger driven reactor, but other concepts are also under development. A key consideration for these units is attainment of fast pyrolysis conditions of short heat-up and cool-down time in order to recover a primary product bio-oil in high yield.

Laboratory studies have confirmed that fast pyrolysis can be achieved in a laboratory scale twin-auger reactor when using a heat carrier medium.¹ More often the auger reactors are reported as char production systems. Slow pyrolysis is well-known as the method for production of char from biomass, which also produces a gas



Figure 1: ABRI-Tech's 1t/d mobile pyrolysis system.



Figure 2: The Agri-Therm field deployable unit (up to 5t/d).

byproduct with condensates including a heavy tar product, "settled tar", and a lower density aqueous phase, "pyroligneous acid." While the focus of Task 34 is fast pyrolysis to produce a single phase primary pyrolysis bio-oil product, the formation of secondary pyrolysis products (gas and two-phase liquids) may also be of interest to our readers. In this short review we will highlight several of the current development efforts under consideration by the Task participants.

Fransham and Badger² have been at the forefront of this development effort for many years. Their auger systems are now available through two separate companies, ABRI-

(Continued on page 4)

Review of developments *...continued*



Figure 3: Transportable fluid-bed pyrolyser from Virginia Polytechnic Institute and State University.

Tech³ and Renewable Oil International.⁴ They have each built several small demonstration systems which have been operated for short periods of time. Detailed product yields and qualities are not readily available and concerns remain that the operation does not fall into the definition of fast pyrolysis because of longer residence times in the hot zone. A discussion of the ABRI-Tech equipment development effort was published in an earlier issue of this newsletter.⁵

The mobile pyrolysis system of Berutti and Briens was introduced in this newsletter in 2007.⁶ The Agri-Therm unit⁷ is a field deployable unit (up to 5t/d) consisting of annular bubbling fluidised beds; the inside bed is for combustion with internal transfer tubes wherein the biomass particles are heated and then injected into the outer bubbling bed for pyrolysis.

Another transportable fluid-bed pyrolyser from Virginia Polytechnic Institute and State University was also reported earlier in this newsletter. That unit was developed to process poultry litter as a means of disposal and energy recovery. A 1-5t/d unit was designed and constructed in 2008.⁸

The effort at the Eastern Regional Research Center (ERRC) of the

US Department of Agriculture was described in this newsletter just last issue.⁹ A 2t/d mobile processing plant based on the circulating fluid-bed catalytic pyrolysis process, tested in the laboratory at ERRC, is being assembled at the laboratory in Wyndmoor, Pennsylvania. Industrial partners include Siemens, who is designing controls for on-farm pyrolysis systems, and PQ/Zeolyst who is collaborating in the scale-up of the production of the catalyst materials.

A recent demonstration was made of two mobile technologies for biomass pyrolysis at Stevenson, Washington, USA. The Western Renewable Technologies system¹⁰ acquired from Biogreen® involves a heated horizontal helical screw conveyor without a central drive shaft. The screw acts in part as an ablative heater and is electrically heated by resistance. The 1t/h throughput is designed for an 8-h day of operation with a daily maintenance period. The reactor can be operated at a range of temperatures to maximise bio-oil vield, or char or gas as alternatives. The Amaron Energy unit¹¹ is a 0.5t/d rotary kiln

"A key consideration for mobile pyrolysis units is attainment of fast pyrolysis conditions of short heat-up and cool-down time."

pyrolyser which has been optimised for production of bio-oil. Although there is limited data on the operations of these units, the demonstration required collection of data and product analyses to validate the systems and those results will be forthcoming.

A Dutch system, PyroFlash, has recently been in the news.¹² The details of the reactor system are not publically available. The developer, Nettenergy, describes a mobile processing system (2t/d) that makes a range of product liquids, solids, and gas. Although they claim on their website to have a "not slow pyrolysis" process,

(Continued on page 5)



Figure 4: Members of the FarmBio³ consortium pose in front of the 2 metric tonne per day mobile pyrolysis unit which is under construction at ERRC in Wyndmoor, Pennsylvania.

Review of developments *...continued*

based on the product descriptions, this unit would not be a fast pyrolysis system.

The group at Fraunhofer UMSICHT in Oberhausen, Germany, is developing a different concept for a mobile pyrolysis system based on the PYTEC ablative pyrolysis system.¹³ The ablative system, also described in the last issue of this newsletter,¹⁴ would be used in the field for pyrolysis of straw following harvest. The liquid yield from the unit would be collected and transported to a central point for utilisation or further upgrading. The technology is being developed in a laboratory scale unit (15kg/h), and a functional mobile model (100kg/h) is also in development with MBB Fertigungstechnik GmbH. Based on the design calculations at UMSICHT it appears the cost of bio-oil would be about the same as baled straw at the field edge. Further cost savings for bio-oil transportation versus straw bale transport would improve the economics.

A similar ablative pyrolysis system is also under development in Russia. The EnergoLesProm company¹⁵ has a small demonstration plant (50kg/h) for their mobile autonomous technology for processing low grade wood to liquid fuel.

In the UK, the European Bioenergy Research Institute (EBRI) at Aston University is developing a range of mobile pyrolysis units. EBRI currently has a 20kg p/h intermediate pyrolysis mobile unit operating in the Punjab region of India. It is demonstrating an alternative to open field burning by utilising the residues from rice and wheat harvests to produce oil, gas and biochar. This 'Energy Harvest' research project is engaging groups of Indian farmers and villagers who are receiving training for operating the unit themselves (more details later in this issue.)



Figure 5: Design study for a "mobile" flash pyrolysis plant from UMSICHT.



Figure 6: The European Bioenergy Research Institute's Energy Harvest unit in India.

EBRI is also building two 20kg p/h mobile intermediate pyrolysis units – 'Pyrofabs' – which will be operational throughout North West Europe later this year as part of its EU INTERREG IVB project, BioenNW. The units will characterise the suitability of a range of biomass for pyrolysis from Belgium, France, Germany, the Netherlands and the UK. A larger 100kg p/h mobile unit is also under construction and will be located in the West Midlands.

In summary, mobile fast pyrolysis has been under development for over a decade. Numerous small (1-5t/d) demonstration plants have been built and operated. None have since moved to commercial operation for applications to lignocellulosic biomass.

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Review of developments *...continued*

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Use of the Pyroformer[™] in the Energy Harvest Project



Sudhakar Sagi of the European Bioenergy Research Institute, Aston University, UK, offers an insight into Energy Harvest mobile pyrolysis in India Bioenergy that is sustainably produced has the potential to mitigate climate change and to bring about rural development and socioeconomic improvement. The Pyroformer[™] technology based on Intermediate Pyrolysis developed by the European Bioenergy Research Institute (EBRI) at Aston University is being used to convert the agricultural residues that are being currently burnt openly on fields in India. This technology converts the agricultural residues after harvest to useful products such as bio-oil and biochar which in turn could be used for energy generation. The project "Energy Harvest" was conceptualised to be multi-faceted in that apart from meeting rural energy needs and reducing CO₂ emissions, it would galvanise self-reliance, local employment, gender and health related issues in addition to land reclamation. The focus was to meet the energy needs of these services for the rural population and stop open burning.

Intermediate pyrolysis The core of the project is the demonstration unit based on Intermediate Pyrolysis. The process used is depicted as a flow sheet in the Figure 1 below.

The Pyroformer[™] (Pyrolysis reactor) is a coaxial screw system through which the pellets made from different agricultural residue biomass are fed via a feed storage hopper. Pellets are conveyed through the inner screw from one end to the other end of the reactor. The residence time of the pellets can be carefully controlled by the speed of the screw. At the end of the reactor the vapour leaves the reactor through the vapour outlet which will be condensed later in a condenser. The feed pellet that is eventually transformed into char partly falls into the outer screw and partly into the char container. The char in the outer screw is then transported back into the reactor and allowed to mix with the fresh pellets that are fed into the reactor. The char thereby achieves two aims: (i) better heat transfer as the char is already hot and (ii) the char also acts as a catalyst to promote better reactions inside the reactor which help in reduction of tar

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^{11.} Amaron website:



Figure 1: Flowsheet of the intermediate pyrolysis technology for conversion of straw.

formation. The reactor is carefully monitored with respect to the reaction temperature by electrical heaters with precise temperature controls.

One of the products of the process, the pyrolysis vapour, is directed to a quenching unit where it is quenched using diesel as a quenching medium which is recirculated in a closed loop until the required blend of pyrolysis liquid in diesel is reached. Once the blend is reached the blended oil is collected and fresh diesel oil is put into the condenser. The oil collected is used for running the dual fuel engine. Not all the vapour generated can be condensed to oil. The non-condensable gas that cannot be condensed is used to run the engine on dual fuel mode. Alternatively there is also a flaring system as a safety measure.

The process parameters for straw conversion are a temperature of 380°C and a residence time of 3 minutes for the straw pellets inside the reactor. The product yields at these conditions were 34% char, 33% condensable oil, and 33% non -condensable gas (by difference). The yields can be changed by changing the process conditions but then the quality of the products is also affected.

Mobile unit demonstration

As part of the project planning the technology was made mobile so that it could be demonstrated in the villages. The mobile pyrolysis plant is shown in Figure 2. The mobile unit was stationed in the Khuaspura village of the Rupnagar District Punjab state of India. The village has a population of around 3000. The mobile unit was stationed at the community centre, which is operated as a school during the day and as a community centre in the evenings. The centre had not been connected to the mains supply and so the school had no power. The Energy Harvest Project provided continuous power supply to the school during its

demonstration in the village . The unit was demonstrated for a period of two months (September 2013 to October 2013).

The total capacity of the heaters installed on the Pyroformer is 12kW. The motors require additional power to rotate the screws of the Pyroformer and also for the feed hopper. The additional power requirement, including the chiller and the pump for the condenser together with screws, is 2kW. In total the power requirement of the plant is 14kW. However, once the desired temperature for the pyrolysis process is reached the power consumption of the plant is minimal. In simple terms the power required by the plant at the start up is around 14kW and once the set temperature is reached the plant consumes around 3 to 4kW.

The total straw converted during this period was around 700kg. The char was also distributed among the rural women who use firewood for domestic cooking. The feedstock was converted into stable products such as blended oil (pyrolysis oil blended with conventional diesel), biochar and non-condensable gas fractions. Pictures of the products are shown in the Figure 3.

Results

The simple mass balance of the whole process states what goes in and what comes out. For a unit (Continued on page 8)



Figure 2: Mobile Pyrolysis unit in the village Khuaspura.



Figure 3: Feedstock, blended oil and biochar.



Figure 4: FTIR spectra of rice straw and biochar.

mass of biomass such as 1 acre of field generating 2 tonnes of rice straw, we get roughly 660kg of biooil, 680kg of biochar, and the rest by difference is the noncondensable gas fraction from processing the residues through intermediate pyrolysis. The feed was pelletised prior to the pyrolysis process. This was taking place at around 120°C. The pellets produced were very hard and highly densified. This can be described as like a pre-treatment step similar to torrefaction. The density of the straw is 840kg/m³. The feedstock straw has a heating value of 16 -17MJ/kg with a moisture content of roughly 5%. The bio-oil blend of 20% has a heating value of 37MJ/kg. The biochar has a heating value of 20MJ/kg. The pH of the biochar is around 9.

The locally produced bio-oil was utilised not only for producing power by running the engine, but also was distributed to the local farmers for them to run on the small scale leister engines installed on their farms for lifting water required for irrigation. Experiments were carried out in both modes by running the engine on pure diesel as well as with various blend ratios of pyrolysis oil and diesel. This study was required to compare the behaviour of the engine as well as the consumption rates of the fuel. From the results it has been observed that the rate of consumption of the blended fuel was not very different from the pure diesel.

Dual Fuel Engine

The capacity of the installed engine in the container is 20kW (25kVA). The engine used for the demonstration of the Pyroformer technology is a Greaves make G 11 series, water cooled, four stroke, multi cylinder with direct injection compression ignition. The alternator is a state of the art selfexcited, self-regulated and brushless design. AVR controlled voltage gives excellent voltage regulation. The entire genset consists of the engine closecoupled with an alternator along (Continued on page 9)







Figure 6: Chart of the fuel consumption at various blending ratios for wheat straw.

with a control panel, all housed inside a canopy. The canopy is an acoustic enclosure that attenuates the genset sound within statutory limits in India. The control panel is fitted with an electronic controller for display and protection. The dual fuel installed on the engine offers an affordable and efficient means of operating diesel engines utilising both liquid and gaseous fuel. This system allows for operation on natural gas up to a maximum of 75% of the fuel required to maintain the desired speed and load. The dual fuel system operates by blending both diesel fuel and natural gas in the combustion chamber. This is achieved using a fumigated gascharge design, whereby gaseous fuel is pre-mixed with engine intake -air and delivered to the combustion chamber via the airintake valve.

The engine was tested for blends up to 20% with a running time close to 250hrs. The engine was inspected on a regular basis and it showed no signs of deterioration. The engine was also tested via a dual fuel mode of operation by injecting the non-condensable gas fraction. The results showed that we could displace the liquid fuel up to 20%. This could also possibly be increased with an increase in feedstock throughput. The results of the engine testing are depicted in Figures 5 and 6 for wheat and rice straw feedstock respectively. Trials are ongoing with rice straw processing and have processed around 1200kgs. The char produced has been given to Punjab Agricultural University who will be evaluating the fertilising capability of the char in a detailed scientific study and applying biochar to the fields in large scale. The field for the biochar trials at the PAU is shown in Figure 7 below.

Feedback from the villagers

The villagers were all very interested in the whole demonstration and participated actively. The villagers also expressed interest in actively participating if the unit will remain stationed there for much longer. In fact, there were few farmers who were not planning to burn the field from the coming harvest season. We did however observe during the harvest season that 4 to 5 farmers did stop burning the field when rice was harvested. Figure 8 shows pictures taken during the demonstration phase in the village Khuaspura. Since we were providing electricity to the community centre the villagers made use of it for their weekly village meetings. They requested that the project remain in the village, and said they will cooperate with us in every way possible.

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Figure 7: Field for biochar trials at PAUL Ludhiana.

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Figure 8: Community Centre and school being powered as part of the village demonstration.



Lignin pyrolysis in a circulating fluidised bed influence of temperature and char holdup on product distribution



In this project the pyrolysis of Kraft lignin in a circulating fluidised bed was studied. The focus lay on the pyrolysis mechanism and on the influences of temperature and char accumulation in the bed on liquid product yields and composition. The yield and composition of gaseous products and char were also investigated (detailed information in (Franck et al., 2014)).

For the experimental investigations the setup shown in Figure 1 was used. The circulating fluidised bed Miika Franck (pictured), Ernst-Ulrich Hartge, Stefan Heinrich, Joachim Werther, all of Hamburg University of Technology, and Dietrich Meier of Thünen Institute of Wood Research, discuss results from their recent study of influences on the pyrolysis of Kraft lignin

riser was 2m in height and 80mm in diameter. The lignin was fed pneumatically by nitrogen into the pyrolyser. The bed was fluidised with a mixture of nitrogen and steam. Solids were separated from the gas by a primary cyclone and recirculated via a standpipe and a syphon. Char and ash were further separated by a secondary cyclone, before the hot vapours were quenched in a scrubber operated with a water/sodium hydroxide solution. The gases left the scrubber with temperatures below 90°C, and passed through a

demister, a platen type heat exchanger and an electrostatic precipitator.

The gas composition of the permanent gases was continuously monitored. In addition side stream samples were taken just before the scrubber and were analysed offline to determine the oil yield and composition. The oil was analysed by gas chromatography (GC) and gel permeation chromatography (GPC) for oligomeric substances (Franck et al., 2012).

(Continued on page 11)

Lignin pyrolysis *...continued*

During each experiment, the initial bed material (quartz sand) was coated with the char remaining from the pyrolysis process. The bed material (BM) and the material caught by the secondary cyclone (C2) were analysed for each experiment to determine the char content and composition. Therefore after the experiment the reactor was cooled down in an inert atmosphere until its temperature was below 300°C. The bed material cooled down further and was then taken from the reactor at an ambient temperature. Char may influence the process since char itself and the contained alkaline and alkaline earth metals have a catalytic effect (Richards and Zheng, 1991). In a technical application of the process the char might be used as source for process inherent energy by combustion, as the pyrolysis process itself is endothermic (Franck et al., 2011).

In this work the influence of pyrolysis temperature on the product yields and composition was studied in the range between 550 and 700°C. To identify the influence of the char in the pyrolysis reactor Kraft-lignin was continuously fed into the reactor and pyrolysed at 650°C (Experiments B1 to B3, cf. Figure. 4). At different times side stream samples were taken and analysed. For all experiments the feeding rate of lignin was 2±0.3kg/h, the

"In this work the influence of pyrolysis temperature on the product yields and composition was studied in the range between 500 and 750°C."



Figure 1: Experimental pyrolysis plant (80mm riser diameter, 2m height).

nitrogen volume flow for pneumatic lignin feeding was 3.8Nm³/h, the steam mass flow was 12kg/h and the superficial gas velocity at the riser bottom was 5.5±0.3m/s. The nitrogen volume flow through the gas distributor was adjusted to maintain a constant superficial gas velocity at the reactor bottom of 5.5m/s.

RESULTS Solids properties and pyrolysis mechanism

Figure 2 shows BM particles with the char layer. It can be concluded that during the pyrolysis of lignin in the circulating fluidised bed, the lignin makes contact with the bed material. Molten lignin deposits on the bed material form a char layer as a result of the pyrolysis reactions taking place at the particle surface level. This assumption is in good agreement with the softening temperature of lignin of ~120 to 180°C (Nowakowski et al., 2010). The result is that the bed material is finally coated with char. The char layer of the particle in the centre of Figure 2 is fractured. The char (Continued on page 12)



Figure 2: SEM image of bed material (BM) from Experiment B2, particle in the centre showing char coating fracture, scale bar at 40µm.

Lignin pyrolysis *...continued*

layer is not stable when subjected to mechanic stress due to impact on the reactor wall or cyclone wall or collision with other particles. The fragments can be found in the material separated from the gas in the secondary cyclone. It consists mainly of small plate like char fragments and a few primary particles not caught by the primary cyclone.

Effect of char on product yield

The yields of oil, gas and char for the three experiments B1 to B3 with the duration of lignin feeding can be seen in Figure 3. The duration of lignin feeding was 68 min for B1 and 130 min and 117 min for B2 and B3 respectively. During experiment B1 two samples were taken, while in experiments B2 and B3 four samples were taken in a sequence. The yields are plotted against the lignin amount fed into the reactor from startup up to the middle of the sampling period. The char produced is approximately proportional to the lignin amount fed into the reactor, and stays mainly in the reactor as the above mentioned char layer that builds up on the bed material. As the bed material is taken from the reactor after each experiment only the yield of char at the end of the whole feeding can be determined. It can be seen that the char yield is slightly lower for the shorter experiment B1 (less lignin fed into the reactor). The gas yield increases with rising char holdup and the oil yield decreases. It can be clearly seen that the char (with its components) has a strong influence, decreasing the oil yield by 7 to 10%-points for the experiments B2 and B3.

This effect can also be seen in Figure 4 showing the molecular weight distribution of the four oil samples of experiment B2 obtained by gel permeation chromatography. The distribution shifts to smaller molar size with



Figure 3: Effect of accumulated mass of lignin fed into the reactor on the yield of oil, gas and char (single points) for the experiments B1 to B3.



Figure 4: Molecular weight distribution of oil samples from experiment B2.

increasing time and thus an increasing char amount in the reactor.

The cracking of oil and gas to smaller molecules may be caused by the char, the amount of which increases with time in the reactor. However, the alkaline and alkaline earth metals also influence the product yield and composition. Due to the pulping process Kraft lignin contains high amounts of sodium (Continued on page 13)

Lignin pyrolysis *...continued*



Figure 5: Effect of temperature on product yield.

(here 3g/kg) which accumulates in the char having a sodium content of 9g/kg for experiment B2. Effect of pyrolysis temperature on product yield

Figure 5 shows the effect of temperature on product distribution for the three products pyrolysis gas, oil and char. The vapour residence time in the hot zone of the CFB pyrolysis plant was shorter than 1s.

It can be seen that the effect of temperature manifests in the maximum oil yield of 49% at 650°C. With rising temperature the gas yield almost doubles from 17 to 32%, while the char yield decreases from 24 to 13%.

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Renewable wood-based biofuels for shipping: ReShip



Kai Toven of the Paper and Fibre Research Institute (PFI), Norway, and Tony Bridgwater from the European Bioenergy Research Institute, Aston University, UK, introduce us to the ReShip Project



ReShip is a new research project which aims at developing renewable wood-based biofuels for shipping. Alternative sustainable transportation fuels are needed in the transport sector to reduce greenhouse gas emissions. In marine transport, there is a particular need to develop lowsulphur fuel alternatives due to upcoming regulations demanding significantly reduced sulphur content in marine fuels from January 2015.

The overall objective of ReShip is to develop cost-competitive pyrolysis oil based multicomponent fuels which meet the performance requirements of marine diesel engines. The project is led by the Paper and Fibre Research Institute (PFI) in Norway and includes industry partners along the whole value chain, from forest owners to end users in shipping. Research and development partners in the project are PFI, Aston University and the Norwegian University of Science and Technology (NTNU). The project is running from 2013 to

2017 and is funded by industry partners and the Research Council of Norway.

Sustainable transport fuel from wood feedstocks

In Norway, low quality wood fractions like forest residues and energy wood represent a significant potential resource for bioenergy production. This resource is currently not much used due to costly logistics and low profitability in direct combustion. However, cost-efficient logistics can be obtained for such feedstocks by converting the biomass into energy dense pyrolysis oils in decentralised pyrolysis plants.

Crude pyrolysis liquids are not suitable for direct use in diesel engines. Compared to petroleumbased fuel oils, the pyrolysis liquids are acidic, rich in oxygenated functional groups, hard to ignite and of relatively low calorific value. In addition, fast pyrolysis oils contain unstable compounds such *(Continued on page 15)*



Figure 1: ReShip aims to produce multi-component fuels for the shipping industry.

ReShip ...continued



Figure 2: The ReShip concept.

as aldehydes, ketones and carboxylic acids, which may cause increased viscosity, phase separation and changed chemical composition upon storage. Pyrolysis oils can be upgraded directly to transport fuel quality by hydro-deoxygenation, highpressure hydrogenation and catalytic cracking of pyrolysis vapours. However it is a challenge to develop cost effective techniques as rather extensive upgrading is required.

ReShip concept

The ReShip project is based on an innovative approach for producing partly upgraded storage stable pyrolysis oil from wood feedstocks, and for developing pyrolysis oilbased multi-component fuels which meet the performance requirements of marine diesel engines. The project concept is illustrated in Figure 2. If successful, the concept can be adapted to an industrial scale at low cost, as both mild upgrading and fuel blending processes are favourable from a cost perspective.

Key topics which will be addressed in ReShip are bio-oil stabilisation technology, multi-component fuels, and engine testing. The research activities are being led by Dr. ing. Kai Toven, Lead Scientist in Biorefining and Bioenergy at PFI and Professor Tony Bridgwater, Head of the European Bioenergy Research Institute at Aston University, UK.

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"Key topics which will be addressed in ReShip are bio-oil stabilisation technology, multi-component fuels and engine testing."

Application of pyrolysis oil in the OP16 gas turbine - development of a low calorific fuel combustor



Martin Beran and Lars-Uno Axelsson conclude their discussion begun in PyNe Issue 33 - of research and development at OPRA Turbines in the Netherlands





Figure 1: The OP16 gas turbine.

OPRA Turbines develops. manufactures, markets and maintains generator sets in the 2MW power range using the OP16 series of gas turbines. The OP16 gas turbine, shown in Figure 1, is an all-radial design, which provides robustness, reliability and the highest efficiency in its class. A key feature of the OP16 gas turbine is the ability to utilise a wide range of fuels. As part of OPRA's continuous efforts to extend the fuel capability of the OP16 gas turbine, a research and development project was initiated focusing on the utilisation of pyrolysis oil. The overall aim of the R&D project was to develop a combustor suitable for the efficient combustion of pyrolysis oil. The higher viscosity, lower energy density and limited chemical stability of the pyrolysis oil means that it requires different fuel handling in comparison to conventional liquid fuels.

A feasibility study on the use of pyrolysis oil in the OP16 gas turbine was presented in an earlier PyNe newsletter (Issue 33, June 2013). This article presents the continuation of the R&D project, focusing on the design and testing of a new combustor for pyrolysis oil and other low-calorific fuels. This research project was partly sponsored by the province of Overijssel, the Netherlands, as part of the BE2O project.

Combustor for pyrolysis oil

Based on the results obtained from the initial feasibility study a new combustor was developed (patent pending, patent application No. US 2012/0111014). The main features of the combustor are a significantly increased volume of the reaction zone and the use of an enhanced cooling method of the flame tube walls. From the feasibility study it was found that the flame tube film cooling significantly disturbs the combustion process of the pyrolysis oil. Hence it was necessary to develop a new strategy for the cooling of the flame tube wall. This new cooling strategy, together with the use of thermal barrier coating on the flame tube inner wall, allows the surface temperature to be kept relatively high and improves the combustion process of the pyrolysis oil.

(Continued on page 17)

Application of pyrolysis oil in the OP16 gas turbine... *continued*

Results from the test campaign

OPRA's atmospheric combustion test facility, shown in Figure 2, was used for the tests. For these tests pyrolysis oil from pine wood provided by BTG Bioliguids was used. The test campaign focused on the operability range of the new combustor as well as on the effect of different fuel injection strategies. The target for the new combustion chamber was to burn pure pyrolysis oil between 70% and 100% load, which was also achieved. At loads below 70% a mixture of pyrolysis oil and ethanol has to be used. The ethanol is also used for cleaning the fuel system and injectors when the engine is shut down.

During the rig testing several air blast nozzles and pressure nozzles were used. The main purposes here were to investigate the impact of the droplet size on the pyrolysis oil burning process and to define the limits for the maximum allowed droplet diameter as a function of the operating condition. The air blast nozzle produces a fairly constant droplet size independent of the fuel flow, whereas the droplet size for the pressure nozzle shows a strong dependency on the fuel flow. In addition, a pressure nozzle is highly sensitive to abrasion and erosion due to the particles present in the fuel and the high velocity in the small nozzle passages. Therefore, a pressure atomiser is not recommended for pyrolysis oil burning. From the extensive tests performed it was found that the maximum allowed droplet diameter for pyrolysis oil



Figure 2: Low calorific fuel combustor installed in OPRA's combustion test facility.

spray shall be approximately 50% of the maximum allowed droplet diameter for diesel No. 2.

The exhaust gas emissions were measured for several fuels over the complete load range. Table 1 shows the NO_x and CO emissions at full load for diesel, ethanol and pyrolysis oil converted to 15% O₂. Although the low-calorific combustor can burn high-calorific fuels, such as diesel and natural gas, the NO_x emissions will be higher due to the longer residence time caused by the larger combustor volume. To make a fair comparison the emission values for diesel are obtained from a conventional gas turbine combustor. The NO_x emissions from low-calorific fuels are generally lower than from conventional fuels, which can be seen when comparing the ethanol and diesel results. However, pyrolysis oil and diesel show similar NO_x levels. It should be noted that pyrolysis oil contains about 0.4%wt nitrogen that is chemically bounded in the fuel. It

Table 1: Exhaust gas emissions at full load converted to $15\% O_2$. For ethanol and pyrolysis oil the data was obtained from the low-calorific fuel combustor, whereas for diesel the data was obtained from the conventional combustor.

	Diesel No. 2	Ethanol	Pyrolysis Oil
NO _x [g/kWh]	3.9	2.4	3.9
CO [g/kWh]	0.56	0.17	1.1

is produced during the combustion process. If all the nitrogen contained in the fuel is converted to NO_x , this would make up for about 60% of the total NO_x produced during pyrolysis oil combustion. Hence, from an environmental perspective it would be beneficial to decrease the nitrogen content in the fuel.

means that the so called fuel, NO_x,

Conclusions

With the development of the new combustor OPRA has an efficient and reliable solution for the utilisation of pyrolysis oil in the OP16 gas turbine. The new combustor can also be used for other (ultra) low calorific liquid and gaseous fuels such as syngas, ethanol, biogas and waste gas.

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Requirements for transportation of fast pyrolysis bio-oil







From top: Mika Laihanen, Antti Karhunen, and Tapio Ranta

Mika Laihanen, Antti Karhunen, and Tapio Ranta of Lappeenranta University of Technology (LUT Energy) in Finland discuss how best to transport biomass fast pyrolysis bio-oil as production increases

Biomass fast pyrolysis bio-oil (pyrolysis oil) is a renewable liquid fuel which can replace fossil fuels in energy production. In Finland there are currently ongoing projects concerning the production of pyrolysis oil. As this is a new chemical product, transportation requirements and conditions must be taken into consideration.

Pyrolysis oil is a liquid biofuel produced, for example, from biomass or other raw materials that contain carbon. Biomass fast pyrolysis bio-oil is generally acidic, unstable at high temperatures or over long storage periods, highly polar and mainly non-volatile containing a large amount of chemically dissolved emulsified water. Due to acidity pyrolysis oil will be classified as dangerous goods in road, railway and waterway transport. However pyrolysis oil has never been transported on a large-scale in Finland.

Pyrolysis oil requires specific equipment for loading, unloading and handling. All surfaces in contact with the product must be acid proof materials. Suitable materials are, for example, acid proof steel PTFE (Teflon). Acidic products are already transported widely and suitable transport equipment exists.

When transporting dangerous goods, labelling is always required and it is also required for road, railway and waterway transportation of pyrolysis oil. In labelling (Figure 1) the lower UN number 3265 stands for "Corrosive



Figure 1: Pyrolysis oil labelling for transportation.

liquid, acidic, organic, n.o.s." and 80 stands for corrosiveness (8) without any secondary hazard (0). For waterway transport pyrolysis oil will also need IMO classifications.

Table 1 illustrates different transportation methods, typical unit sizes and indicative annual transportation flows in Finland when production of pyrolysis oil is 100,000 tonnes per annum. For calculation purposes, the hypothesis is that all pyrolysis oil will be transported regularly using a single transportation method; usually, in practice, the logistical chain could consist of combinations of different alternatives. For railway transport it is assumed that one train consists of ten chemical wagons and tank containers are transported by road with semi-trailers. Maximum payloads per unit can vary between different European countries.

Suitable transportation methods are always closely related to production sites and end-users' facilities, existing transport connections and annual production

(Continued on page 19)

Requirements for transportation *...continued*

volumes. Transport distance is also a very important factor.

Road transportation with a truck and full-trailer combination is currently the most cost-effective and flexible option if no railway connection or deep-water harbour exists near the production plant. The main advantages of road transport are the constant transportation flow from production to end-users and that roads offer the most extensive transport network.

The advantage of railway and waterway transport methods is the option of larger unit sizes per one transport. Railway transportation would be a good option for straight long-distance domestic routes from production plant to end-user; however, if extra loading and unloading are needed the profitability of railway transport could decrease.

Waterway transport with chemical tankers is the best method for large -scale exportation when the size of

Table 1: Different transportation method unit sizes and number of annual transports in Finland with a production of 100,000 tons p/a.

Transportation Method	Maximum payload per unit, tonnes	Number of transports per year
1. Roadway	36-42	~2,500
2. Railway (10 wagons)	540	~200
3. Waterway	3,600*	~30
4. Tank container	25-30	~3,500

*volume of one tank in chemical tanker 3,000m³

one load is at least 3,000m³. Waterway transportation also requires chemical ports and sufficient storage capacity to operate. In Finland truck transport is often needed in some part of railway and waterway transport logistical chains.

Requirements and classifications for pyrolysis oil transportation are needed because large-scale production should be commercialised in Finland within the next few years. Pyrolysis oil will be classified as dangerous goods in transportation but it can be



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Figure 2: Truck and full trailer combination for chemical transports in Finland.

Chemical fingerprinting of pyrolysis oil heavy ends with high-resolution mass spectrometry



The chemical complexity of pyrolysis oils poses a challenge for their characterisation at the molecular level. The heavy ends of pyrolysis oils (i.e. polar fraction of high molecular weight species) are difficult to characterise using conventional gas chromatography

Janne Jänis of the University of Eastern Finland looks at the best way to characterise pyrolysis oil at the molecular level

based techniques. This fraction contains a large number of nonvolatile polymeric compounds, e.g. pyrolytic lignin and different cyclic, polyhydroxy types of compounds ("sugaric" compounds).

In recent years, many types of analytical techniques have been utilised for chemical fingerprinting of pyrolysis oils, such as conventional gas chromatoghraphy (GC), comprehensive twodimensional gas chromatography (2D-GC), infrared spectroscopy (IR), nuclear magnetic resonance spectroscopy (NMR) and highresolution mass spectrometry (HRMS).¹ GC is superior in identification of small volatile organics but generally does not work with high-MW species. IR and NMR provide information about functional groups but do not generally allow identification of individual compounds from the mixture.

HRMS has been succesfully used in the past for the characterisation of petroleum and its end-products.² (Continued on page 21)



Figure 1: High-resolution FT-ICR spectrum of birch wood fast pyrolysis oil. About 1400 individual heteroatom compounds could be identified. The inset shows the magnified view (a half nominal mass unit) at around m/z327. All nine baseline-resolved peaks could be unequivocally assigned to different oxygen-containing species, with varying degree of aromaticity/unsaturation (DBE = 1-11).

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Chemical fingerprinting of pyrolysis oil *...continued*



Figure 2: 3D van Krevelen plot of all compounds identified from the phase-separated pine wood slow pyrolysis oil by FT-ICR MS: aqueous phase (left), oily phase (right). A colour coding of the dots represents the relative abundance of each detected species.

Even the heaviest parts of petroleum (e.g. distillation residues), having thousands of species, can be analysed by HRMS techniques.³ More recently, HRMS has also been applied for the analysis of pyrolysis oils from different starting materials. HRMS provides a chemical fingerprint of pyrolysis oils' polar compounds without chromatographic separation. Large polymeric species can also be detected. Currently, the combination of 2D-GC and HRMS looks most promising for detailed molecular level characterisation of pyrolysis oils.¹ The biggest drawback of HRMS is that it is merely semiquantitative as the peak intensities do not correlate with the absolute concentrations of analytes in an oil sample. Relative quantitation between different samples for a given analyte class are, however, possible. Also, some of the smallest analytes (methanol, acetic acid etc) are not typically efficiently ionised, so a combination of GC-MS with HRMS is therefore desirable for more comprehensive analysis.

We have recently used ultrahighresolution Fourier transform ion cyclotron resonance (FT-ICR) MS, combined with different selective ionisation techniques, for compositional analysis of different pyrolysis oils. Figure 1 shows the FT-ICR mass spectrum of birch wood fast pyrolysis oil; about 1,400 unique heteroatom compounds. mainly oxygenated species, could be detected.⁴ Figure 3 shows 3D van Krevelen diagrams (H/C vs. O/ C ratio, relative abundance given by the colour) for the compounds detected from the phase-separated pine wood slow pyrolysis oil by FT-ICR MS.⁵ A van Krevelen plot is a straghtforward visual means for differentiating between lipids, phenolic and "sugaric" compounds in pyrolysis oils. As can be seen, the oily phase has clearly higher content of lipids while "sugaric" compounds are concentrated in the aqueous phase.

In the near future, we wish to combine our FT-ICR MS with 2D-GC technique to obtain deeper insights into the molecular complexity of different pyrolysis oils. In addition, we will test the analytical capabilities of ion mobility spectrometry combined with mass spectrometry (IMS-MS) in pyrolysis oil characterisation. IMS-MS should futher advance our understanding of pyrolysis oil constituents, especially for different isomeric species.

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Second generation bio-oil by selective decarbonylation via catalyst-impregnated pyrolysis



Paul Dauenhauer of the University of Massachusetts Amherst, USA, discusses a new method of increasing the viscosity and commercial value of bio-oil

High-temperature pyrolysis of lignocellulosic biomass produces gases, solid residue and condensable vapors which can be converted to biofuels and biochemicals.¹ The vapours, referred to as 'bio-oil,' are comprised of a large number of highly oxygenated chemical species, some of which have multiple functional groups and are extremely reactive. Over periods of weeks and months, these species react to form heavier oligomers, which increase the viscosity and reduce the overall value of bio-oil.

Implementation of the thermochemical biorefinery concept relies on the capacity for



Figure 1: Tunable cellulose melt (liquid) chemistry by catalyst-impregnated pyrolysis converts reactive furans (e.g. HMF) to stable furans (e.g. methylfuran).²

bio-oil to be produced in sufficient quantity and quality that it can be traded on the open market as a commodity. This approach severs upstream pyrolysis and downstream refining processes, where the latter is accomplished with an approach similar to today's centralised petroleum refineries. For pyrolysis oil to become a commodity, it must be produced to have sufficient energy density and stability to be valuable and be able

> "We introduce a new technology called Catalyst -Impregnated Pyrolysis"

to be transported over reasonable distances via pipeline. Despite the need for higher quality bio-oils, the technology and scientific understanding needed to accomplish this are lacking, especially with regards to controlling pyrolysis chemistry to enhance selectivity to desirable products (stable bio-oil).

We introduce a new technology (Cat. Sci. & Tech., 2014), called Catalyst-Impregnated Pyrolysis,² for tuning the reaction pathways of melt-phase pyrolysis of cellulose and lignocellulose to targeted products more desirable for the second generation of bio-oil. As shown in Figure 1, the global pathways of cellulose pyrolysis have been defined as transition from solid polymers, through a liquid melt rich in anhydrooligomers, and the eventual formation of high-vapour-pressure (Continued on page 23)

Second generation bio-oil *...continued*



Figure 2: Pyrolysis oil yield versus selectivity to decarbonylated furans. Pd/C achieves almost 90% selectivity to stable furans with only negligible loss in bio-oil yield.²

species which evaporate and are collected as bio-oil.³ Within the melt, condensed-phase reactions break down biopolymers into nonvolatile anhydro-oligomers which ultimately form volatile species. The composition of evaporated species is dictated by melt-phase reactions, and controlling them is one approach to producing a more stable and energy dense bio-oil.⁴

Impregnation of biomass feedstocks with supported metal catalysts enables the tuning of melt -phase reactions as melt species have sufficient mobility (and residence time) to interact with catalyst active sites. In this work, we target one class of suspected "bad actors" – aldehydes and ketones – which can destabilise bio -oil. Figure 2 shows that selective transformation (decarbonylation) of aldehydic furan species at 500°C is accomplished with Pd and Pt supported on Al₂O₃, SiO₂ or carbon. Additionally, decarbonylation occurs without a significant reduction of total bio-oil yield. Pt-containing catalysts exhibit a strong capacity for C-C cleavage, which produces more permanent gases and significant loss in the overall pyrolysis oil yield. However, Pd-containing catalysts only minorly impact overall yield, with only a small increase in permanent gases. Moreover, Pd achieves almost 90% selectivity to carbonyl-free furans including furan, methyl furan, and dimethylfuran.

While catalyst-impregnated pyrolysis with Pd provides a high

impact proof-of-concept of the potential of the technology for the next generation of bio-oils, the approach opens the door for future research necessary for implementation. The future programme of research must identify multiple chemical targets for *in situ* melt-phase transformation, synthesise cheaper and more recoverable catalysts, and develop continuous flow pyrolysis reactors capable of impregnating, reacting, and recovering catalysts.

Support

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Production of liquid feedstock via catalytic fast pyrolysis of biomass in the presence of steam



Efthymios Kantarelis of KTH in Sweden looks into the potential of steam for the biomass fast pyrolysis process

The use of metal modified zeolite catalysts for production of liquid feedstock and a modified reaction environment (using steam) during biomass fast pyrolysis was investigated. The introduction of steam and the incorporation of metal functions on the zeolite (multifunctional catalysts) can promote in situ hydrogen generation (steam reforming, WGS, dehydrogenation and decarbonylation), hydrogen transfer and hydrogenation reactions which are important to produce olefins and aromatics as well as hydrogenated products of high market value.1,2

Fast pyrolysis process

A continuous bubbling fluidised bed reactor was used to pyrolyse a mixture of pine and spruce samples in the presence of different catalysts. The pyrolysis unit is composed of five sections: the gas preheating section, the feeding section, the fast pyrolysis reactor, the char–vapour separation section and the liquid quenching and collection system. The overview of the process is shown in Figure 1. The pyrolysis took place at 450°C with a steam to biomass ratio (S/B) of 0.5 with the experimental duration at 90 minutes.

Catalysts

Silica supported Ni and V were prepared by incipient wetness impregnation and tested for their activity and effect on the liquid yield and composition during fast pyrolysis of biomass. The metallic functions were then incorporated into a hybrid catalyst comprising of H-ZSM5 zeolite and bentonite as binder. The different effects on *(Continued on page 25)*



Figure 1: Overview of fast pyrolysis process. H-401 Steam Boiler; H-402 and H-403 Gas preheaters; R-201 Fluidised bed reactor; V-101 Biomass hopper; SF-101 Metering screw feeder; SF-102 Injection Feeder; FG-201 Cyclone; V-301 Venturi Scrubber; P-301 Pump; E-301 Heat Exchanger.

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Production of liquid feedstock *...continued*

liquid yield and composition were investigated by adjusting the metals to zeolite and binder ratios as well as the Weight Hourly Space Velocity (WHSV). Characterisation of the coke formed, assessment of its reactivity during regeneration, and evaluation of the structural stability of catalyst, were all carried out.

Results

The effects of the different catalysts on the CHO composition of the liquid are shown in Figure 2. Straight lines represent the theoretically obtained composition (in terms of H/C and O/C) if dehydration (-H₂O), decarbonylation (-CO) or decarboxylation (-CO₂) were the only acting mechanisms of oxygen rejection (NC refers to non-catalytic pyrolysis). The figure shows that Ni is more active in de-oxygenation (lower (O/C) than V; however, liquid with H/C content lower than that obtained from the V-catalysed pyrolysis is obtained. This effect is detrimental because hydrogen scavenging results in low effective hydrogen content (EHI), which will make further upgrading difficult due to excessive coking.³ V seems to selectively deoxygenate liquids by decarboxylation and decarbonylation reactions. The Ni-V bimetallic system shows enhanced deoxygenation activity compared to V-containing catalysts, while simultaneously showing less hydrogen scavenging compared to Ni catalysts. The H/C and O/C atomic ratios of the obtained liquid show that deoxygenation moves along the theoretical dehydration pathway up to 50% of ZSM5 loading. The dehydration is catalysed by the strong Brønsted acid sites and is the rate limiting step for Diels-Alder cyclisation and aromatics production via the production of olefins.⁴ The increased zeolite content allows for olefins cyclisation and di-, tri- and oligomerisation reactions after olefins



Figure 2: Effect of different catalysts on the CHO composition of obtained liquid (dry basis).

production. Increased catalytic activity (at low WHSV) results in oxygen rejection in the form of carbon oxides.

XRD patterns of the fresh and regenerated catalyst are shown in Figure 3. Their examination indicates no new phases, nor changes in the relative intensities, while the analysis of interplanar spacing, 'd', shows that the catalyst retained its crystallinity and thus, it can be said that no significant structural alterations occurred.

Conclusions

- Both selected metals (Ni and V) are catalytically active in transforming the composition of pyrolysis liquids. The H₂ concentration in the produced gases increased. Vanadiumcontaining catalysts are more selective in the reduction of carbonyl-containing compounds (mainly acids and ketones). Both metals showed activity towards the production of phenols.
- Increased zeolite content

reduced the yield of liquid product which had significantly lower oxygen content. However, increased zeolite content results in increased coke formation on the catalyst. The metal/zeolite content did not significantly affect the char yields.

- Low WHSV favour oxygen rejection as carbon oxides. However, lower liquid yields were obtained, while the char yield remained almost unaffected. The coke percentage on the catalyst is reduced.
- Comparison of reactivity of coked catalysts at different WHSV showed that at low WHSV more reactive coke is deposited on the catalysts which denotes that the main deactivation mechanism is coke formation inside the pore structure.
- The XRD and microscopic investigation of the regenerated catalyst at low temperatures (550°C) did not show significant structural defects,

Production of liquid feedstock *...continued*

which suggests that the catalyst is hydrothermally stable.

 Overall, the modified reaction environment and bimetallic (Ni-V) multifunctional catalysts based on ZSM5 zeolites promote hydrogen production and transfer within the reaction system to produce a deoxygenated liquid with preserved H content.

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Figure 3: XRD patterns of fresh and regenerated catalyst (WHSV ½ h-1). (a) Fresh catalyst; (b) regenerated at 550°C (1st row). SEM and TEM microphotographs of fresh (2nd row) and regenerated catalyst (3rd row).

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EMPYRO: Realisation of a commercial scale, biomass fast pyrolysis plant



Gerhard Muggen of BTG-BTL updates us on the work of Empryo in the Netherlands



The outline of the Empyro project was presented in PyNe News Issue 27 (June 2010). While it has taken more time than anticipated the key objective of the Empyro project remains unchanged: to demonstrate the fast pyrolysis technology on a 5t/hr capacity and make use of pyrolysis oil to replace natural gas.

Fast pyrolysis plant

The Empyro plant will be situated in Hengelo on the site of chemical company AkzoNobel, and in April site specific construction work began. All permits have been obtained for the realisation and operation of the plant on this site.

The feedstock is clean wood and long term biomass contracts could be concluded to guarantee the biomass supply. Final drying will (Continued on page 28)



The Empyro Pyrolysis Plant



Figure 1: Impression of the Empryo fast pyrolysis production plant.

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EMPRYO ...continued



Figure 2: Schematic drawing of the boiler to be installed at FrieslandCampina in Borculo (left). On the right, the Stark double register burner applied in the boiler.

take place on site, and the biomass dryer has been engineered by German company Amandus Kahl. This company will also supply and install the unit. The engineering of the pyrolysis plant itself was performed by BTG-BTL together with Zeton. Stork and HoSt. In the plant 5 tonnes of dried biomass will be converted hourly into about 3.2 tonnes of pyrolysis oil. Excess heat generated from the combustion of the byproducts (gas and char) will be used for the generation of steam. Subsequently, this steam will be used to provide the heat for the biomass dryer and to run a steam turbine to generate electricity. The excess steam will be supplied to AkzoNobel (located next to Empyro) for salt production. Due to the efficient design of this plant a total of up to 90% of the energy in the biomass will be captured in the form of pyrolysis oil, steam and power while no external energy will be required to run the plant (apart from start-up). To get a sense of the scale of this installation: the framework alone weighs 225 tonnes while the largest single component weighs another 52 tonnes; the height of the plant will be close to 24 metres.

The pyrolysis plant is being constructed by Zeton, Enschede. A benefit of working with Zeton is that they assemble and mechanically test the main processes of the complete Empyro plant at their inhouse workshop before partially dismantling the installation into skid mounted modules and transporting these to site. This will also save

"Due to the efficient design of this plant a total of up to 90% of the energy in the biomass will be captured in the form of pyrolysis oil, steam and power while no external energy will be required to run the plant (apart from start -up)." time as site preparation work can be done in parallel to plant construction. Zeton will also be in charge of re-assembly on site, which is actually only a few kilometres from their own premises.

Pyrolysis oil application

Obviously, an extremely important part of the realisation of the project is a guaranteed long-term demand for pyrolysis oil. FrieslandCampina one of the world's largest dairy companies - owns and operates a number of natural gas fired boilers for the generation of process steam. Agreement has been reached on a 12 year delivery contract for the majority of the pyrolysis oil, which FrieslandCampina will utilise as a substitute for natural gas to generate 40t/h of 20 bar(g) process steam for use in milk powder production. A big advantage of this application is that every pyrolysis oil/natural gas substitution ratio up to 70% is possible with a 100% redundancy of natural gas. Therefore, the co-firing of pyrolysis oil will not affect or interrupt the core business of

(Continued on page 29)

EMPRYO ...continued

Figure 3: Due to the innovative nature of the technology, Empryo can cover almost 40% of the total investment using European, national and local grants.

FrieslandCampina. Through the use of pyrolysis oil, the direct CO_2 emissions for the entire Borculo location - one of the largest production facilities of the company - will be reduced by approximately 15%.

For these purposes, a new boiler has been designed by Stork. Construction of the boiler has recently begun, and it is scheduled to be online in the first quarter of 2015. Instrumental in the design have been the pyrolysis oil firing tests performed jointly by Stork and BTG-BTL (see PyNe News Issue 31). These have given insight into the NO_x, CO and dust emissions of pyrolysis oil as well as the fouling behavior when fired using Stork's standard Low NO_x Double Register gas and oil-burner. The pyrolysis oil will be transported from the production site in Hengelo to FrieslandCampina in Borculo by tank truck, which is just a 30km distance. Locally, use will be made of a 100m³ storage tank.

Project financing

The total investments for design, construction and initial start-up of

the Empyro plant are around 19 million Euros. Due to the innovative nature of the technology, Empyro was able to cover almost 40% of this investment by European, national and local grants. More specifically, financial support was obtained from the European Commission (FP7 Programme), the Ministry of Economic Affairs via the Topsector Energie TKI-BBE Programme, the province of Overijssel, and the Energy Fund of Overijssel (EFO). Additionally, equity was provided by EFO and a private investor from Enschede.

Time schedule

Both site work and construction have started. It is expected that the Zeton skids will be ready to be transported to site in October and reassembly will begin at that time. Mechanical completion is expected to be reached before the end of 2014. Commissioning and start-up is scheduled for Q1-2015. In the same period the construction and commissioning of the new boiler of FrieslandCampina will be completed, and ready to start firing pyrolysis oil. Contact Gerhard Muggen BTG-BTL P.O. Box 835 7500 AV Enschede The Netherlands

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In-line determination of the water content of pyrolysis oils

Franz-Peter Girke discusses the latest research and developments in the multistep "bioliq® concept" at Karlsruhe Institute of Technology (KIT) in Germany

Motivation

An important step in KIT's multistep "biolig® concept" for synthetic fuel production from dry lignocellulosic biomass is the mixing of slurries from the pyrolysis products (oil and char) which are converted downstream in the entrained flow-gasifier to synthetic gas. As a long-term objective of this work process improvements on the relevant technologies need to be achieved with regard to safety technology (e.g. the detection of phase separated volumes) and process control (e.g. by a fully automated feed control for an optimal oxygen dosage to the gasifier).

In previous test campaigns on the new pilot plant the pyrolysis products were analysed labour intensively in the laboratory. However, significant simplifications can be achieved by using wellchosen and adapted high frequency technologies for process analytics.

Research efforts

Concerning the analyses of BioSyncrudes[®] (high-concentrated suspensions of pyrolysis condensates and char) taking samples is very problematic. The composition of small sample volumes for the purposes of analysing a process medium is usually different to the composition of the medium inside the process. Furthermore, in lab analyses, different operating conditions (pressure, temperature, etc.) exist to those in the real process and therefore any transfer of lab results to real conditions can lead to significant errors. All in all, time-

"In previous test campaigns on the new pilot plant the pyrolysis products were analysed labour intensively in the laboratory. However, significant simplifications can be achieved by using well-chosen and adapted high frequency technologies for process analytics."

Figure 1: Microwave Transmissions and Reflection cell from *hf-Sensor*.

shifted and costly lab analyses are undesirable. Hence, real-time and easy analyses without sample handling are desirable, which provide direct information about the actual composition of the process medium. During the process, *in situ*, inline measuring methods ideally with real time results - are required.

Measuring technology

According to preliminary measurements, a special microwave measuring technology was suitable for integration into a pipeline construction in order to detect the water and char content of pyrolysis-oil char suspensions.

Microwaves with a frequency of approximately 1GHz up to approximately 10GHz are well suited to displace polar substances into oscillation. This effect can be used for measurement purposes. Higher frequencies cause strong losses to occur from relaxations of the molecules, while at lower frequencies the conductivity influence of the substance rises. Therefore, the use of microwaves in the region of 1GHz to 10GHz seems to be the best method to determine water content of pyrolysis oils.

For measurements like this metallic pipes form a so-called "circular waveguide" in which transmission modes of microwave signals are injected in an ingenious way into the pipeline, and consequently also into the medium inside the pipe as (Continued on page 31)

In-line determination of water content *...continued*

Figure 2: *Left:* Transmission curves; *Right:* Reflection curves of the pyrolysis oil samples (y-axis arbitrary units, x-axis microwave frequency in MHz).

longitudinal and transversal waves. Depending on the dielectric properties of the medium the microwaves are absorbed, transmitted and/or reflected. Microwaves have the advantage of penetrating non-destructively into

"The first focus of the microwave measurements was to determine the water content of the samples. As a reference the Karl-Fischer-Method was selected as a reference for the real water content." the material to be examined and no warming effects are expected on the test material because the inserted microwave energy is much too small. Moreover, a sufficient penetration depth of the microwaves into the bulk material under test allows the detection of chemical components further away from the sensor, so this measuring technology is well-suited for online or inline analytics.

The microwave measuring cell of the company hf sensor (Leipzig, Germany) being used operates within a microwave frequency range of 2.0GHz up to 3.0GHz and was developed especially as a transmission and reflecting cell (see Figure 1). It has been specially adapted for measurements under high pressure, for narrow internal pipe diameters, and for the difficult insertion of microwave signals into the pipe. For batch measurements the microwave tube was filled with the medium and then closed, and the temperature was adjusted. After attaining the desired temperature, several measurements were carried out on the material samples with this configuration.

The first focus of the microwave measurements was to determine the water content of the samples. The Karl-Fischer-Method was selected as a reference for the real water content.

Results

The emitted microwaves are absorbed, transmitted and reflected in a substance. Transmission and reflection do not necessarily depend on each other. Insofar that transmission spectra and reflecting spectra can deliver independent information, it appears to be reasonable to consider and combine both types of spectra for *(Continued on page 32)*

In-line determination of water content *...continued*

Figure 3: Predicted water content (via PLSR method) versus the reference water content (by Karl-Fischer titration).

statistical evaluations.

By means of the

"ÚNSCRAMBLER X10.2" software of the "CAMO" company (Oslo, Norway) the measured spectra have to be analysed as a data matrix. Because of the complicated curve progression it is not possible to evaluate them manually. These recorded curves must be assigned to the samples and the transmission and reflection data is combined in one single data matrix after average centred weighting of the values.

First a PCA (Principal Component Analysis) is carried out. This allows a first overview of possible relationships between the different pyrolysis oils. From the ample original spectral data, PCA delivers a compressed data matrix, which can be used for the classification of pyrolysis oils and also for development of a correlation model between the microwave spectra and the water content. By the use of three such Principal Components more than 95% of the data variance is explained. If a PLSR (Partial Least Square Regression) is applied, a good correlation between reference value and measured value of the water content can be obtained by the common processing of transmission data and reflecting data.

The predicted exactness of the water content calculated by the PLSR model is fairly accurate. The maximum divergence of approx. 6wt.% appears at very high and at very low water concentrations. Though the standard laboratory method of Karl-Fischer titration is more precise and well validated, the water measurement by means of microwave spectroscopy as an inline method can show changes in the water concentration very quickly during production and handling of pyrolysis oil.

Contact

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Pyro2014

Marianne Bell and Scott Banks, both of the European Bioenergy Research Institute at Aston University, UK, review

Delegates listening to one of the 70 presentations at Pyro2014.

The 20th International Symposium on Analytical and Applied Pyrolysis, organised and chaired by Tony Bridgwater of the European Bioenergy Research Institute (EBRI) at Aston University, took place from the 19th – 23rd May 2014, in the centre of Birmingham, UK.

The week-long conference was universally considered a great success, with 230 delegates from 32 countries around the world. Across the week, they heard from 70 presenters delivering the latest findings in topics including analytical pyrolysis; applied pyrolysis; pyrolysis kinetics and mechanisms; and catalysis in pyrolysis, together with research results from a huge number of posters.

Themes and presentations Several themes emerged from the conference relevant to the future of pyrolysis, with one of the most important being the increasing relevance in this area of catalysts. The inclusion of catalysts is becoming a major part of pyrolysis research due to their ability to enhance various compounds produced during the process. A large proportion of Pyro2014's oral presentations featured research that made use of catalysts, and eight were specifically focused on the topic of catalyst use itself in two dedicated sessions.

Another key point that emerged from the conference was that waste has become a more mainstream feedstock for pyrolysis conversion to either a fuel or a chemical source. This was in particular highlighted by presentations by Frank Riedwald, "Waste printed circuit board

(Continued on page 34)

Pyro2014 ...continued

Pyro2014 delegates at the Gala Dinner on 23rd May 2014.

pyrolysis with simultaneous sinkfloat separation of glass and metals by contact with a molten salt – a laboratory investigation", and Sam Haig, "Fuels from waste plastics: an independent review and economic assessment of current technologies", among others.

Finally, perhaps the most unusual feature of the conference was the focus on pyrolysis uses outside of the typical thermal chemical conversion technique. Presentations focused on bacterial identification (Kent Voorhees, "Catalytic pyrolysis for bacterial identification", carbon nanotube production (Aidan Smith, "A novel process for hydrogen and carbon nanotubes production from the pyrolysis-gasification of waste plastics"), and many other innovative topics, particularly among the large number of poster presentations.

Posters and prizes

Pyro2014 benefitted from three poster sessions spread across the week, and also featured its own

poster competition, with presentations for the winners taking place at the gala dinner on the fourth night of the conference. The winning poster was by Wolfram Buss, from the University of Edinburgh, entitled "Biochars affected by re-condensation of pyrolysis vapours - investigation of phytotoxic effects and characterisation of re-condensed products". Frontier Laboratories also awarded prizes for knowledgeable and enthusiastic presentations, with first prize awarded to Guillermo Rosas Mayoral, from the University of León, for his poster "Auto-thermal and mobile pyrolysis reactor for vineyard residues with low pretreatment".

Sponsors

Pyro2014 received great support through sponsorship from: ERDF; BioenNW; CDS Analytical; Shimadzu; Gerstel; Aston University; Frontier Lab; Gas Data; PyroLab; SciMed; SUPERGEN Bioenergy Hub; and Elsevier, without which the conference could not have taken place.

Pyro2016

The 21st International Symposium on Analytical and Applied Pyrolysis will take place in 2016 at CRNS Nancy in France. We wish all of the Pyro 2016 team all the best for the organisation and delivery of another successful Analytical and Applied Pyrolysis conference.

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www.aston.ac.uk/eas/research/ groups/ebri/

For more detailed information on the conference proceedings, please visit www.pyro2014.co.uk

International Events

News Flash: Pyrolysis Workshop

BRISK will be holding a fast pyrolysis workshop which is open to all to attend, organised by Wolter Prins and Tony Bridgwater. Subject: Review of challenges and opportunities in biomass fast pyrolysis processes

Location: Ghent, Belgium

Date: December 8th-9th 2014

AUGUST 2014

24th-28th Lignin 2014 Umeå, Sweden

26th-28th 2014 China Guangzhou International Biomass Exhibition Guangzhou, China

SEPTEMBER 2014

2nd-5th <u>TCS 2014: Symposium on Thermal and Catalytic</u> <u>Sciences for Biofuels and Biobased Products</u> Denver, Colorado, USA

2nd-4th <u>4th International Symposium on Gasification and</u> <u>its Applications</u> Vienna, Austria

8th-11th <u>ISWA 2014 Solid Waste World Congress</u> Sao Paulo, Brazil

10th-12th <u>12th International Chemical and Biological</u> <u>Engineering Conference</u> Porto, Portugal

15th-18th Bioenergy from Forest Marina Congress Centre, Helsinki, Finland

16th-19th <u>18th World Congress of CIGR</u> Beijing, China

21st-22nd GMEE2014 Hong Kong, China

21st-23rd <u>4th Annual World Congress of Bioenergy</u> Qingdao, China

SEPTEMBER 2014 continued

24th-25th Biofuels International Conference Marriott Hotel, Ghent, Belgium

30th-2nd <u>7th European Forum for Industrial Biotechnology</u> <u>and the Biobased Economy</u> Champagne-Ardenne, France

Further details will be announced shortly on the

BRISK website.

Alternatively, email i.i.watkinson@aston.ac.uk for

further information.

OCTOBER 2014

5th-11th XXIV IUFRO World Congress 2014 Salt Lake City, Utah, USA

13th-15th <u>33rd International Conference on Thermal</u> <u>Treatment Technologies & Hazardous Waste</u> <u>Combustors</u> Baltimore, Maryland, USA

15th-16th 8th SGC International Seminar on Gasification Malmö, Sweden

20th-22nd Argus Biofuels and Feedstocks 2014 London, UK

26th-29th Lignobiotech III Concepción, Chile

NOVEMBER 2014

17th-20th <u>Renewable Energy from Waste Conference 2014</u> San Jose, California, USA

17th-20th <u>5th International Symposium on Energy from</u> <u>Biomass and Waste</u> Island of San Servolo, Venice, Italy

19th-21st <u>RENEXPO®</u> South East Europe Bucharest, Romania

For further international events, visit www.pyne.co.uk.

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Pyrolysis Related Events

Umeå Plant Science Centre warmly welcomes everyone to the international conference "Lignin 2014" in Umeå, Sweden, August 24 -28 2014.

The Lignin 2014 conference aims to strengthen our knowledge on the various aspects of lignin biosynthesis and utilisation, from the basic cell biology, molecular biology to pre-treatment of plant biomass and utilisation of lignin. The conference is organised with the support of the Formas-financed strong research environment BioImprove and the strategic research environment Bio4Energy, supported by the Swedish government.

- The sessions of the conference are: 1) Cell and developmental biology
- of lignin
- 2) Lignin monomer biosynthesis and transcriptional regulation
- 3) Lignification: Oxidative polymerisation of phenolic monomers
- 4) Diversity of natural lignins
- 5) Modification of lignins in plants
- 6) Lignin characterisation
- technologies
- 7) Lignocellulose processing and extraction of lignin
- 8) Novel applications for lignin and lignin derivatives

Contact

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For questions regarding hotel bookings or travel, please contact: Elisabet Norlin <u>elisabet@umea-congress.se</u>

090-13 00 35

www.Lignin2014.se

Symposium on Thermal and Catalytic Sciences for Biofuels and Biobased Products

September 2-5, 2014

Denver, Colorado, USA

Tcs2014 brings together leading scientists and engineers to share the latest advances in thermal and catalytic science for biofuels and biobased products

<u>Topics</u>

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

www.tcs2014.org

Pyrolysis Related Events

-2014 organised by and of San Servolo . Venice . Italy 7-20 November 2014

venue

The production of energy from alternative sources and its impact on climate change are among the main strategic tools implicated in the sustainable development of our society. Numerous types of biomass and wastes contribute towards the production of energy and reduction in the use of fossil fuels by means of biological, chemical and thermal processes. Existing biomass and waste to energy

technologies are currently undergoing rapid development. Despite growing interest in the use of these technologies, in many countries their implementation remains limited.

The aim of the Venice 2014 Symposium is to focus on the advances made in the application of technologies for energy recovery from biomass and waste and to encourage discussion in these fields. The previous edition of the Symposium, held in 2012, was attended by nearly 580 scientists and operators from approximately 62 different countries.

The fifth edition of the Symposium will be held in the stunning island of San Servolo in the Venetian Lagoon and will feature:

- Three days of scientific presentations ٠
- One day of guided technical tours at biochemical and thermochemical • plants
- Six parallel oral sessions (one of which in Italian), poster sessions and • an exhibition by companies working in the field
- Expected attendance of over 600 delegates from tens of different countries worldwide

The Symposium is organised by the International Waste Working Group (IWWG) with the scientific support of the Universities of Queensland, Padova, Hokkaido, Rostock, Trento and Hamburg University of Technology.

Publications

Fuel - Volume 116

Published by Elsevier Publication date: January 2014

Within this journal is the following paper: <u>Effects of CO2 on biomass fast pyrolysis: Reaction rate, gas yields and char reactive</u> <u>properties</u> Authors: C Guizani, FJ Escudero Sanz, S Salvador

Journal of Analytical and Applied Pyrolysis - Volume 107 Published by Elsevier Publication date: May 2014

BRISK Newsletter - Issue 4 Published by Aston University Publication date: May 2014

The Selection Process of Biomass Materials for the Production of Bio-fuels and Co-firing The Selection Process of Biomass Materials for the Production of Bio-Fuels and Co-Firing (IEEE Press Series on Power Engineering) Published by Wiley-IEEE Press Publication date: June 2014 Author: Najib Altawell

EERA Bioenergy NEWS - Issue 3 Published by Aston University Publication date: June 2014

ISBN: 978-1-118-54266-8

Publication date: June 2014

Transformation of Biomass: Theory to Practice Published by Wiley Publication date: September 2014 Edited by Andreas Hornung ISBN: 978-1-119-97327-0

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Further Information

If you require further information about the PyNe newsletter, or you would like to contribute to future editions, please contact the Editor:

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Past editions of PyNe newsletters are available on the website.

www.pyne.co.uk

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IEA Bioenergy Agreement Task 34 Newsletter - PyNe 35