IEA Bioenergy
Technology Collaboration Programme
Task 34

Newsletter
Direct Thermochemical Liquefaction

Stay all safe and healthy until we meet again

PyNe 47
Jan 2021

Inside this Issue:

This time we welcome you with a fresh look of the PyNe newsletter in accordance with the new IEA Bioenergy corporate design that was introduced in 2020. This new design will also be implemented on our website; not unlike many other things these days we do see some delay here but hope to get that realized early 2021 now that all required backend changes are set.

Most of you will not be surprised that we continued work in the task in a virtual manner just like the majority of the world in face of the CoVid-19 pandemic. The fall meeting 2020 was intended to take place in the U.S. connected with the TCS 2020 at Washington State University; we also planned a workshop on material compatibility in conjunction with that meeting. As many of you may know, TCS 2020 took place online and I must say that this was a very pleasant experience for me – well organized, many excellent presentations, and vivid discussions despite the comparably short term change to a digital format.

Task 34 met online several times instead of meeting in the U.S. However, we decided to postpone the workshop on material compatibility to 2021 to allow for finding a suitable format. This workshop is dedicated to exchange experience gathered with suitable materials for FPBO and biocrude processing. There has been quite some work on it – also shown by the feature article chosen from PyNe 28 ten years ago: ‘Assay of corrosion resistance of two selected metals exposed to bio-oil’. Please make sure to follow our updates not to miss it; we will announce well ahead.

We all hope to meet again in person during fall 2021 but also learned that there is little room for planning, yet. And while writing itself is not affected too strongly by the current pandemic, many other issues require our attention and/ or limit availability worldwide. This lead to realize that we need to publish a somewhat shorter than usual PyNe issue 47.

It contains the second part of the strategy for valorizing pyrolytic lignin obtained from water extraction of FPBO developed by the Green Chemical Reaction Engineering group at University of Groningen/ The Netherlands.
This part features the evaluation of oxidative and reductive strategies for this specific fraction.
IEA Bioenergy Task 34 has conducted another Round Robin, this time focussing on suitable methods for quantification of heteroatoms in FPBO oils and also biocrude from HTL. The article has been published already and we will present a summary of its main findings here.
Finally, the advantage of using a semi-batch reactor to analyse kinetics of hydrothermal biomass fractionation is shown with results from a cooperation of University of Hohenheim and KIT/ Germany.

As promised we kept up the work to supply you with latest DTL information. We published a report on ‘Commercial status of direct thermochemical liquefaction technologies’ showcasing 20 commercial and demonstration DTL plants around the world (https://task34.ieabioenergy.com/dtl-commercialization-report/).

We also published a detailed case study on the use of FPBO to supply high temperature heat in industry as part of a larger inter-task project of the IEA Bioenergy (https://itp-hightemperatureheat.ieabioenergy.com/). I highly recommend having a look at these examples, especially if you feel to need more information after this comparably short PyNe.

Yours sincerely,

Axel Funke

Task Lead and NTL Germany

Towards a pyrolysis biorefinery: Valorization strategies for the pyrolytic lignin fraction (part II)

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Pyrolysis liquids obtained by fast pyrolysis of biomass are thermally unstable and prone to repolymerization due to their chemical heterogeneity and acidity. Therefore, efficient upgrading strategies are necessary to further convert them into valuable products. An attractive concept is the so-called pyrolysis-based biorefinery, where all fractions of the pyrolysis liquids are valorized separately, preferably with highly carbon-efficient technologies. One interesting approach within this concept is an initial fractionation of pyrolysis liquids by simply adding water, yielding a sugar-rich (pyrolytic sugar – PS) fraction and a hydrophobic lignin-derived (pyrolytic lignin – PL) fraction.[1] These two fractions can be processed independently into a wide range of valuable intermediates and products by strategies tailored to their nature and inherent properties. Unfortunately, the valorization of the PL fraction is relatively unexplored due to its complex structure and low concentration of monomers.[2] This untapped potential has motivated us to further investigate depolymerization strategies to convert PL into added-value products. Following previous insights regarding the catalytic hydrotreatment of PL (part I of this article [3]), we here discuss an oxidative strategy for PL depolymerization using ozone.

Lignin-derived structures are particularly prone to oxidation due to the presence of aromatic rings with high electron density. Ozone is considered an attractive oxidizing agent, as it can be easily generated in-situ from oxygen by an electrical discharge (technology available from lab to industrial scale). Its high oxidizing power allows for a non-catalytic process that can be carried out at mild, ambient conditions, facilitating operation and downstream processing.

In our first exploratory study, pine-derived PL was dissolved (33 wt%) in methanol (MeOH) and exposed to ozone in a semi-batch reactor. Analyses of the oxidized PL showed a molecular weight reduction of up to 40%, resulting in a product oil with much higher volatility. A range of low molecular weight dicarboxylic acids (DCAs) and esters were identified in yields up to 45 wt%, along with highly oxygenated, larger molecular weight aliphatic structures (see Figure 1).[4] DCAs are valuable intermediates for various applications in the polymer and food industries and are currently produced

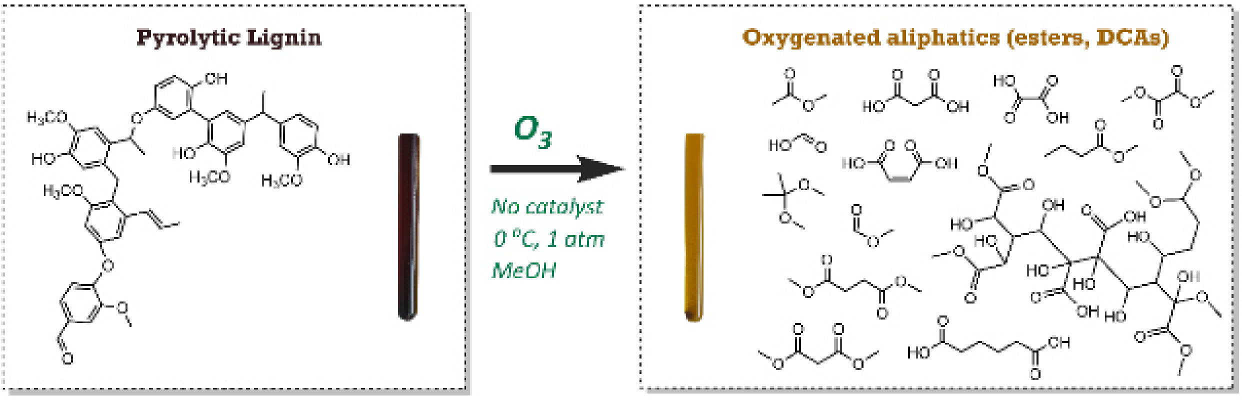
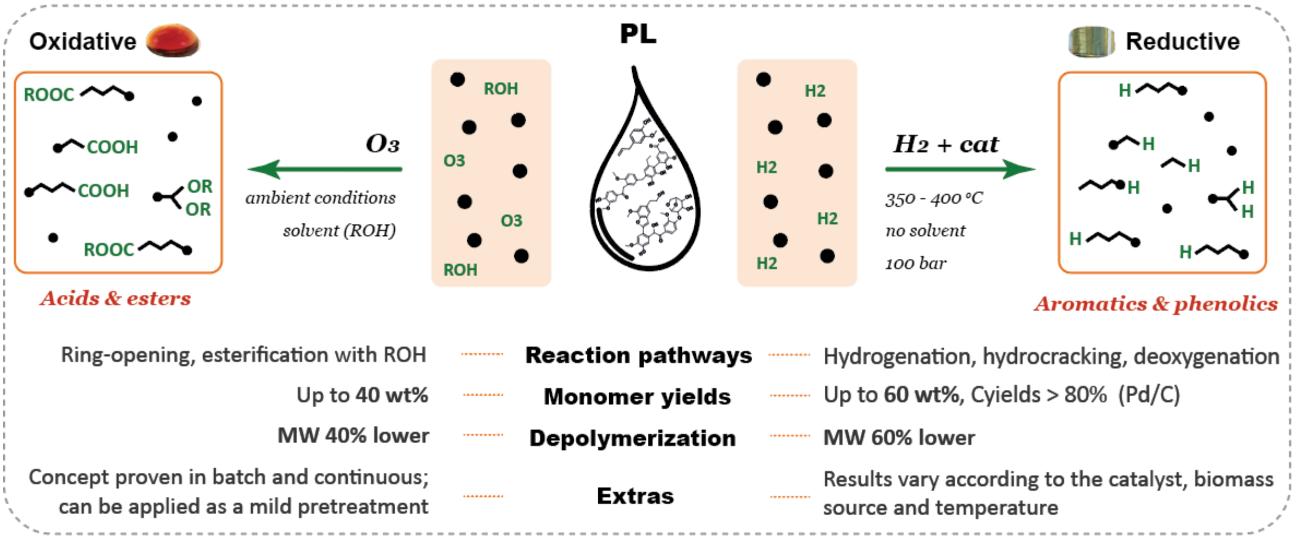


Figure 1: Overview of PL oxidation using ozone.[4]

This study also indicated that PL depolymerization occurs mostly via ring-opening pathways and unsaturated bond cleavage involving ozone, and that secondary oxidation and esterification reactions (in which MeOH participates) broaden the product spectra. Further experiments using lignin model compounds showed that the presence of hydroxy substituents in the aromatic network is the main responsible for such high ozone reactivity. In a follow-up project, the scope of ozone oxidation was explored using insoluble, more condensed technical lignins of higher structural complexity (such as Kraft and Alcell), which were successfully depolymerized in an ozone-mediated solvolysis performed at ambient conditions with both MeOH and EtOH. [6]

Inspired by the promising results and mild conditions required for the depolymerization of PL using ozone, we have successfully performed ozone oxidations of PL in a continuous flow microreactor. The microreactor technology is considered particularly advantageous when considering the enhanced mass and heat transfer rates, safety, and energy efficiency. We observed rapid depolymerization (seconds time scale) of lignin when performing ozonation continuously in this set-up, combined with a lower consumption of ozone and less complex reaction mixtures due to a reduction in the rate of secondary reactions.[7] Finally, we have explored a two-step strategy in which mild ozonation serves as simple pretreatment for PL before catalytic hydrotreatment, aiming to boost PL depolymerization at the expense of some aromaticity (minimized by short ozonation times).[7,8] The oxidative lignin pretreatment had positive effects on the subsequent hydrotreatment reaction, yielding product oils with improved properties and composition. Accordingly, the analyses showed low oxygen content, significantly lower M_w, higher monomer yields, higher volatility, and improved calorific values compared with oils from the direct hydrotreatment of PL.

Our main findings when evaluating oxidative and reductive strategies for PL valorization are summarized in Figure 2. Overall, the two studied routes lead to very different product mixtures, with the potential to be valorized for different applications. While the results reported so far are certainly promising, there is a lot of room for exploration and optimization on the way towards a feasible and well-integrated pyrolysis-based biorefinery.
We hope that our research will support and stimulate future developments in this area. Further studies will be focused on typical process-related aspects like reactor and process design and techno-economic analyses, as well as product-related aspects like detailed product-property analyses and selection of the most profitable outlets for the obtained compounds.



Ring-opening, esterification with ROH	**Reaction pathways**	Hydrogenation, hydrocracking, deoxygenation
Up to **40 wt%**	**Monomer yields**	Up to **60 wt%**, Cyields > 80% (Pd/C)
MW 40% lower	**Depolymerization**	**MW 60%** lower
Concept proven in batch and continuous; can be applied as a mild pretreatment	**Extras**	Results vary according to the catalyst, biomass source and temperature

Figure 2: Overview of our main findings regarding PL ozonation and catalytic hydrotreatment.

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Acknowledgments

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Results of the International Energy Agency Bioenergy Round Robin on the Analysis of Heteroatoms in Biomass Liquefaction Oils

Bulsink, P.; de Miguel Mercader, F.; Sandström, L.; van de Beld, B.; Preto, F.; Zacher, A.; Oasmaa, A.; Dahmen, N.; Funke, A.; Bronson, B.

The latest Round Robin conducted by IEA Bioenergy Task 34 has been published [1]. The focus of this Round Robin was on determination of heteroatoms in biomass liquefaction oils (BLO), specifically nitrogen, sulfur, and chlorine. Heteroatoms play an important role for direct combustion applications of BLOs. Fuel nitrogen and sulphur lead to NO_x and SO_2 emissions, which in turn are subject to many regulatory frameworks. This leads to regulation of heteroatom content in fuel, one important example being the International Maritime Organization (via MARPOL Annex VI) which has reduced the allowed sulfur content in fuels from 3.5 to 0.5% and to 0.1% in Sulfur Emission Control Areas, as of 2020 [2-4]. Trace chlorine analysis is needed for determining the potential for chloride stress corrosion and materials compatibility in equipment.

This fact becomes even more important considering the prevalence of austenitic stainless steel in bio-oil and biocrude handling, upgrading reactors, and potential biomass insertion points into refineries [5-7]. Moreover, sulfur and chlorine are also well-known catalyst poisons while nitrogen imposes an additional catalytic burden during heteroatom removal used to remove oxygen in upgrading bio-oils and biocrudes [8,9].

This latest Round Robin was designed to be a screening test for different methods – the laboratories were free to choose which method to apply and how. Recommendations were given only on how to treat the samples and conduct sub-sampling. Consequently, the results can be used for guidance of further development to establish reproducible determination of above-mentioned heteroatoms. Also, for the first time, biocrudes from HTL were included in the Round Robin to test differences to fast pyrolysis bio-oil (FPBO) in determining parameters with the same set of methods.

Carbon, hydrogen, and water content

These are by now well-established analytical methods for FPBO analysis. They were included to check general laboratory practice and also to investigate applicability to HTL biocrudes. Reproducibility was generally in line with previous studies, also for HTL biocrudes. One notable observation was that relative standard deviation for determination of the water content in HTL biocrudes was comparatively high due to the low water content of the samples. It may be that methods for water content in HTL biocrudes could be better optimized for the sample type/ lower water content, or that laboratories struggled with the analysis of HTL biocrudes due to the higher viscosity as compared to FPBO. Feedback showed that not all laboratories followed sample handling instructions and this fact requires more attention in future. It was concluded that ASTM E203 could need further refinement for HTL biocrudes to improve reproducibility of water content determination.

Nitrogen

Nitrogen content needs to be determined according to ASTM D5291 and also needs to be reported for FPBO as boiler fuel to follow EN 16900 even though this standard does not set a limit. The recommended methods state a limit of quantification (LOQ) of 1000 mg kg^{-1}, which is close to the nitrogen content of many BLOs; especially for those produced from feedstocks with low nitrogen content such as woody biomass. This fact is reflected by the observation from the latest Round Robin: reproducibility of nitrogen determination is good for BLOs from nitrogen-rich feedstocks but drops for those produced from feedstocks with low nitrogen content.

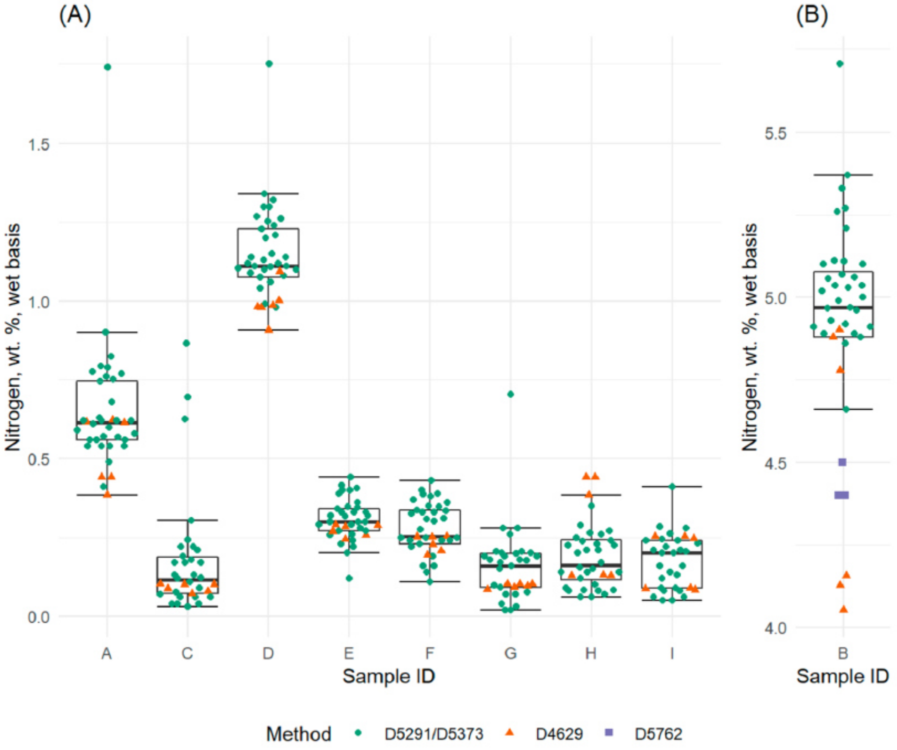

Figure 1: Nitrogen content of BLOs analyzed by 14 laboratories using three methods [1].

Methods that enable lower LOQ (e.g. ASTM D5762 and D4629) were applied by some laboratories and it was found that they require further development. The solvents chosen in these methods have been developed for petroleum based products and are not suitable for BLOs. It was concluded that an additional inter-laboratory study is required for those methods that allow for a LOQ<1000 mg kg^{-1} given that there is need from regulatory frameworks.

Sulfur

ASTM D7544 requires pyrolysis oils to have a sulfur content under 500 mg kg^{-1} to be used in industrial boilers. However, it prefers the use of ASTM D4294 for which certified BLO standards are not available. All laboratories consequently applied a different method for sulfur determination. Amongst the variety of methods test, a preferred method could not be identified and there may be more than one suitable approach. However, reproducibility is a concern, especially given the limit set by ASTM D7544 or international marine fuel sulphur specifications. Additional work is needed to identify which method appears most promising and then complete an inter-laboratory study focussing on the chosen method.

Chlorine

Similar to sulfur, various methods were employed by the participating laboratories for chlorine determination with a high level of variability between datasets. Of all the methods employed, microcoulometry was suggested as a key method that could be further targeted. Microcoulometry performed reasonably well, provides a low LOQ (1–5 mg/kg) and it is a one-step method, reducing risk of operator or handling error compared to two-step methods such as the bomb calorimeter/ion chromatography method.

However, this method detects all halides and is not specific to chlorine. A future inter-laboratory study focussing on the microcoulometry method to assess its suitability is recommended from the work.

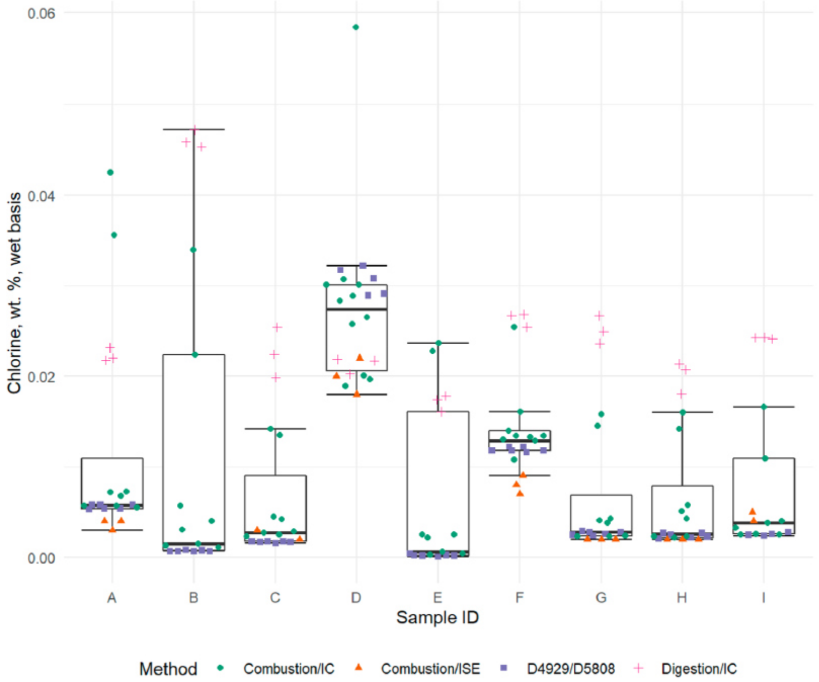

Figure 2: Chlorine content of BLOs analyzed by eight laboratories using four methods [1].

Sampling issues

The samples included a blind duplicate from an FPBO and it was observed that this duplicate significantly deviated in all analyses except hydrogen content. This suggests that the subsamples sent to the laboratories were not equivalent and that correct sub-sampling remains an issue. The challenges of sub-sampling may be even more pronounced for BLOs with high viscosity (such as HTL biocrudes), BLOs from ash rich feedstocks, and BLOs with high solids load.

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More insights into hydrothermal biomass fractionation: Using a semi-batch lab reactor

David Steinbach [1], Ursel Hornung[1], Andrea Kruse[2], Jörg Sauer[1]

Most lab studies investigating biomass conversion, like direct liquefaction or pretreatment, are carried out in small batch reactors which are easy to construct and to operate. However, during biomass conversion the primary products are often not stable under reaction conditions and are subject to consecutive reactions. Therefore, at longer reaction times those primary products are no longer present in the product mixture. An additional issue for batch studies is, the larger the batch reactor is, the longer it takes to cool down the content to room temperature after the desired residence time. During cooling period, consecutive reactions still take place, which has an unwanted effect on the accuracy of kinetic measurements.

To investigate the formation of instable primary products during solid biomass conversion, it is worth spending the effort to build up a semi-batch lab reactor. This reactor type allows to combine rather long reaction times for the low-reactive solid biomass and a short reaction time for the reactive primary products. The concept is to place the solid biomass as a fixed bed in the reactor and to continuously flow the liquid phase through the reactor. In this way, soluble primary products from biomass conversion are continuously removed from the hot reaction zone and thus protected as far as possible from secondary reactions.

Depending on the sampling and analysis frequency of the product liquid, a lot of information can be generated from one biomass conversion experiment. In contrast to this, batch investigations require a lot of experiments to determine the residence time dependence.

For biomass conversion using homogeneous catalysis this semi-batch concept provides an additional advantage. On one side the catalyst can be added to the liquid input stream after the biomass-containing reactor reached the desired reaction temperature. And on the other side, the catalyst dosage can be stopped, when the reaction time is passed.

We firstly studied pretreatment of lignocellulosic biomass with the 100 mL semi-batch reactor, as shown in Figure 1. In the following an example of spruce wood hydrolysis with sulfuric acid is given. The formation of soluble compounds like sugars, furfurals, and organic acids depending on the reaction time of solid biomass is shown in Figure 2.

The reaction time of solid biomass was in total 2 h. If that time would be applied in a batch reactor, primary products of biomass hydrolysis, like xylose from the hemicellulose structure, would completely undergo degradation reactions. A residence time distribution experiment was performed with the semi-batch reactor loaded with biomass. At a flow rate of 15 mL/min the mean residence time of the liquid phase in the reactor was 7.0 min. So, the residence time of liquid in the reactor is one order of magnitude smaller than that of solid biomass.

Figure 2 shows the formation curves of the main hydrolysis products of spruce wood. Cellulose hydrolyzes slowly to glucose at 180 °C under the applied concentration of diluted sulfuric acid, which can be recognized by the long release time of glucose. At 200 °C, cellulose is completely reacted after 2 hours and at 220 °C this hydrolysis time is shortened to 40 minutes. On one side, the hemicelluloses are stable to a great extent under hydrothermal conditions at 180 °C during reactor heat-up, because the release of xylose and mannose increases only

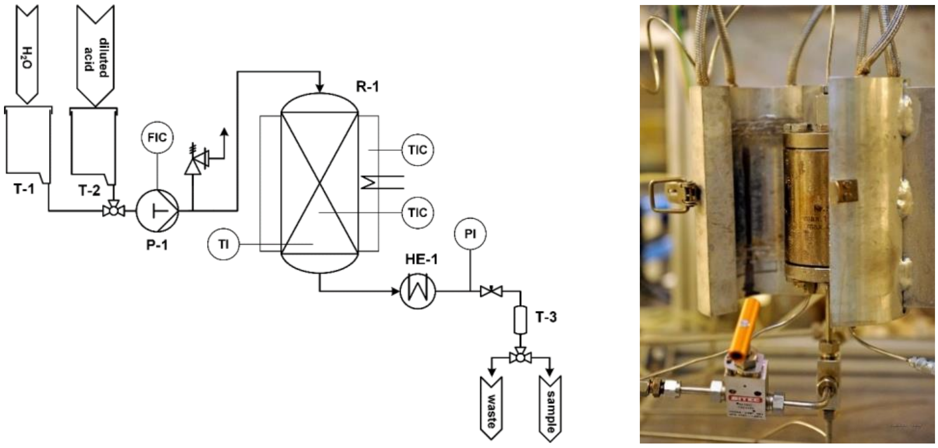

Figure 1: Process flow diagram of the semi-batch test rig (left) and 100 mL fixed bed reactor clamped into an aluminum heating jacket equipped with six heating elements (right)

after the addition of the acid catalyst (see Figure 2). On the other side, the hemicelluloses are largely hydrolyzed during hydrothermal heat-up to 220 °C, which is shown by the declining concentrations of mannose and xylose. From the formation curves of the main hydrolysis products during conversion of lignocellulose, it can be concluded that HMF, levulinic acid and formic acid derive from hexoses, whereas furfural is a secondary product of xylose.

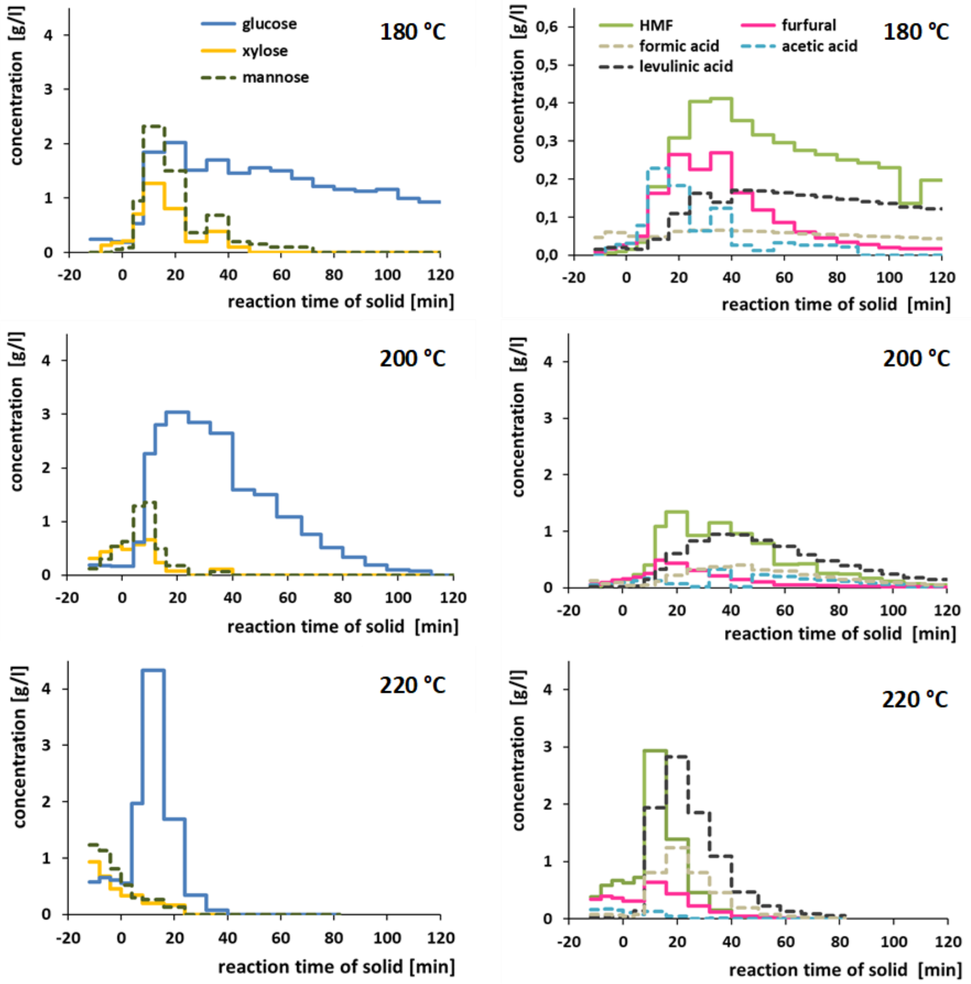

Figure 2: Formation of monosaccharides (left) as well as furfural, hydroxymethylfurfural (HMF) and organic acids (right) during the hydrolysis of 14.6 g spruce wood chips with 0.05 mol/L sulfuric acid at 15 mL/min flow and 25 bar at different temperatures, *t* = 0 min marks the beginning of acid hydrolysis, heat-up is performed hydrothermal without acid catalyst

At the moment the semi-batch lab reactor is used for the delignification of lignocellulosic biomass at organosolv pulping conditions using a methanol-water solvent with phosphoric acid. A consecutive catalytic reduction to aromatic chemicals will then be performed in a fixed-bed reactor, which is currently being designed.

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What happened 10 years ago?

It is interesting to see how the field of direct thermochemical liquefaction developed over the years. We are thus presenting one example highlight from the PyNe newsletter twenty years ago in this regular feature...:

Assay of corrosion resistance of two selected metals exposed to bio-oil

A synopsis from Rolf Strenziok (pictured above) and Helen Rickhoff from the University of Rostock, Germany

Two materials were tested in different concentrations of fuel and bio-oil by using exposure tests at raised temperature, in a liquid- and vapour-phase region (Figure 1).

1. Experimental setup with the two materials and the ageing oven

As reference fuel(s) E85 (85 vol% Ethanol + 15 vol% ASTM -C) was chosen. In addition three different mixtures of E85 and pyrolysis oil with weight ratios of 2:1, 1:1 and 1:2 were used. The tested material samples consist of an aluminium alloy (AlMgSi1), which is often used in fuel pumps and lines, as well as a lamellar grey cast iron (GG25) which is used widely in cars, pumps, compressors etc.

2. Results

In Figure 3 the macroscopic visible changes of the grey cast iron sample in different fuel mixtures after 13 weeks of exposure are shown. While there were no signs of corrosion detectable in the reference fuel, all other samples showed an extensive decomposition, which seemed to increase in higher concentrations of bio-oil. This could not be verified by determining mass loss (Figure 4), because of the hardly removable, black coating on the surface especially in the vapour phase area, which was also increasing in higher concentrations of oil. This coating might have a preventing effect on the corrosion attack and has to be analysed in further tests.

The microscopic observation and the determination of mass loss showed that the corrosive damage on the aluminium sample seemed to be less distinctive. Under the light microscope numerous areas with damage to the passive layer were found. A metallographic cross section (Figure 2) of one of the samples with the highest concentration of bio-oil showed pitting attacks with a depth of at least as 230 µm after 13 weeks. The pitting corrosion is a far more

Continued on page 12

Figure 1: Experimental setup exposure tests

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You can access the full article by using the following link: https://task34.ieabioenergy.com/wp-content/uploads/sites/3/2016/10/IEA-Bioenergy-Task-34-PyNe-28-newsletter-v2.pdf

Upcoming Events

20^{th} Jan. 2021

Workshop 5: Social impacts of woody biomass

Online

SAVE THE DATE 20 January 2021, 15.00 -18.00 CET Registration for the series of online workshops devoted to exploring REDII implementation and delivering good woody […]

https://www.ieabioenergy.com/blog/ieaevent/workshop-5-social-impacts-of-woody-biomass/

21^{st} Jan. 2021

IEA Bioenergy Webinar – Integration of Biogas Systems into the Energy System

Good day, IEA Bioenergy invites you to participate in a free international webinar entitled, “Integration of Biogas Systems into the Energy System” presented by Jerry […]

https://www.ieabioenergy.com/blog/ieaevent/iea-bioenergy-webinar-integration-of-biogas-systems-into-the-energy-system/

28^{th} Jan. 2021

Hydrothermal Liquefaction (HTL) in the Green Energy Transition

Online

Webinars with moderated panel debate and audience Q&A The European Commission’s bold ambition to become the first climate neutral continent by 2050 and achieve a

https://www.ieabioenergy.com/blog/ieaevent/hydrothermal-liquefaction-htl-in-the-green-energy-transition/

25th Apr. 2021

http://www.pyro2020.org/

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in the IEA Bioenergy ExCo
to assess the benefits of joining
Task 34.
https://www.ieabioenergy.com/
directory/executive-committee

IEA Bioenergy Task 34 Website

www.task34.ieabioenergy.com

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Past Issues of the Task 34 Newsletters

http://task34.ieabioenergy.com/iea-publications/newsletters/

If you would like to contribute an article to the Task 34 newsletter or have questions, please contact:

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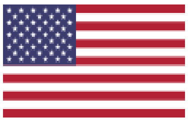
Task 34: Direct Thermochemical Liquefaction



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