BL2F
Transforming Black Liquor to Biofuel

Research and Innovation Action
H2020-LC-SC3-2019-NZE-RES-CC

D7.5 - Report on the Summer School

WP7 - Task 7.2
July 2023 [M33]

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Disclaimer

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Abbreviations and acronyms

Acronym	Description
BL	Black Liquor
BL2F	Black Liquor to Fuel
HTL	Hydrothermal liquefaction

Executive Summary

The Report on the Summer School presents the program and outcome of the summer school for young scientists and Phds organised in M33 of the project (13th and 14th June 2023). This report summarises the organisation, proceedings and results of group work of the Summer School, and preliminary findings from the post-event survey.

Keywords

Black liquor, summer school, intermediates, sustainable fuel

1 Introduction

1.2 Summer School Objectives

The objective of the BL2F Summer School is to disseminate the results and idea of the BL2F project to young scientists and Phds from the partner organisations as well as external participants. The idea is to educate young scientists and Phds about current strategies and pathways to renewable fuels and to put this in relation to the principle process chain of the BL2F procedure. This is essential since future renewable fuel production will be performed in dedicated plants producing one or a narrow range of fuels used close by (de-centralized production) due to a variety of different resources and technologies. Therefore, refinery type plants are going to utilize various renewable, stable intermediates from scalable technologies depending on availability and market in future. Following intermediates are the most promising and were the main topics of the BL2F Summer School:

- Bio-oil / -crude from thermal biomass treatment like HTL and Pyrolysis
- Methanol from syngas
- Hydrocarbons (e.g. via Fischer-Tropsch) from syngas
- Methane from digestions or CO_2-hydrogenation

These intermediates can be produced and traded all over the world (where renewable energy and energy carriers are cheap) to be imported and adding value by their conversion into fuels and chemicals. In this context a pulp mill is the best example to address integrated sustainable fuel production beside paper production and chemical recovery.

The goal of the summer school was to inform and educate the participants, including PhD students and young professionals, about various intermediates, including the biocrude from BL2F. It aimed to facilitate discussions on the challenges related to different process routes, empowering them to develop and exchange their own ways for selected intermediates.

The summer school exchanges brought benefits to current researchers, allowing them to showcase their latest research to younger generations. It was also an opportunity for the researchers to incorporate the knowledge and perspectives from the future generations into their research. By doing so, we recognized that the participants are the next generation of leaders in this research field and would play a vital role in shaping what is to come.

This mutual exchange of ideas and insights enriched the learning experience for all participants and fostered a collaborative environment for advancing the field of study.

2 Implementation of the summer school

2.1 Organisation of the BL2F Summer School and proceeding

The core workshop organisation team was made up of the partners including KIT, LGI and TAU with scientific contributions to the program in form of talks and discussion from TAU, KIT, PSI, VTT, Valmet, NESTE and Ranido at the event.

KIT and TAU planed the scientific program. LGI created and published the visuals on social media, coordinated the registration web page and accompanied the event with a photo documentation and organized a survey. All partners helped to disseminate the visuals.

The proceeding of the summer school consists of scientific talks to the different intermediates and uses the chance that many other working groups at KIT are investigating other intermediates than HTL biocrudes. This gave the possibility to perform various labvisits to all above mentioned intermediate recoveries. Over all a balanced program between BL2F project partners and KIT was established. Seven contributions are from BL2F partners and five are from other KIT working groups.

Appreciating the Finnish tradition of the coordinator and comparable to the first project meeting the social event at the evening of the first day of the summer school was held on a lake with catering and option to swim.

The highlight of the summer school was a group work of the participants, who had the task to research a process chain that deals with one of the mentioned intermediates in order to gain sustainable fuels. The results of the group work were presented to the audience.

2.2 Target audiences

The main target groups of the BL2F Summer School were young scientist and Phds from academia and industry (including catalyst developers, chemical engineering manufactures, refinery specialists).

2.3 Promotional Materials:

Save-the-Date visual and link to registration: a graphic was designed to pique interest in the workshop and was disseminated widely on the BL2F social media, with the first post sent out in February 2023.

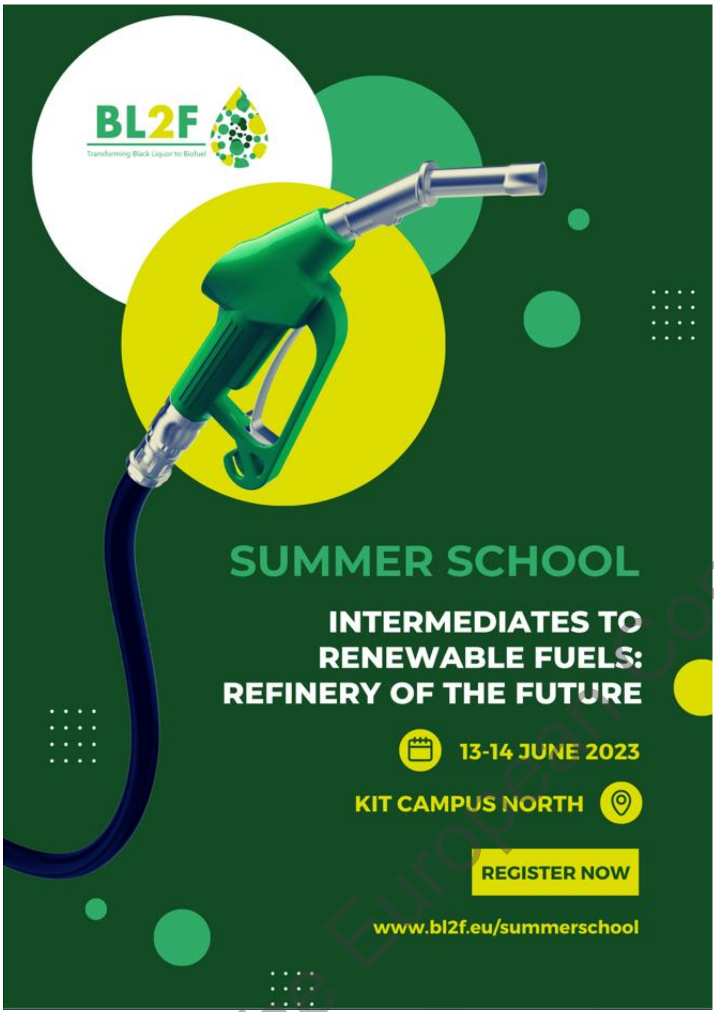

Figure 1: Save-the-Date visual and registration

Poster: a poster with the final agenda was designed and disseminated before the Summer school.

An email was sent to related projects and graduation schools to inform them and invite them to register to the Summer School. The BL2F partners were also encouraged to disseminate the promotional material to their contacts and interested stakeholders.

A leaflet with program, maps and and additional information was prepared for all participants (Annex I)

3 Workshop Proceedings

The summer school was held on 13 & 14 June 2023 at KIT Campus North as face to face meeting. Two online presentations were given, a key note lecture from Valmet by Tooran Khazraie and the presentation of the coordinator of BL2F Tero Joronen on the production of bio-oil integrated to Pulp Mill.

3.2 Day 1

Tuesday 13.6. 2023 **Building 727, Room 202**	
9.00	Registration
10.00	Intermediates to renewable fuels Motivation and goal of the summer school (Nicolaus Dahmen, KIT)
10.30	High Temperature Liquefaction: Status and an example of pilot demonstration (Jukka Konttinen, TAU)
11.00	On the fundamentals of supercritical water salt separation and concomitant HTL using black liquor (David Baudouin, PSI)
11.30	Break
11.50	Lab visits at KIT-IKFT | VERENA (Hydrothermal Gasification), Python (fast pyrolysis), Continuous HDO, Continuous HTL, Mosys and catalyst testing (DME/OME synthesis)
13.00	Lunch
14.00	Hydrodeoxygenation (HDO) of bio-oils (Juha Lehtonen, VTT)
14.30	Oxymethylene ethers (OME) as clean and sustainable diesel substitutes (Marius Drexler, KIT)
15.00	Hydrogen economy and use for transport fuels Solid carbon products from CO2 – theory, practice, products and markets (Jukka Konttinen, TAU)
15.45	Reactor concepts for synthetic natural gas production from CO2 (Mathias Held, KIT-EBI)

3.3 Day 2

Wednesday 14.6.2023 Building 141, Room 735	
9.30	Towards liquefaction of sustainable feedstock to upgraded intermediate products (Tooran Khazraie, Valmet)
10.00	Pyrolysis oils and their applications (Frederico Fonseca, KIT)
10.30	Group work on case studies for different intermediates
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14.00	Tour to KIT Energy Lab 2.0 – Plant network linking electric, thermal and chemical energy flows as well as new information and communication technologies
16.00	Distinctive aspects of techno-economic analyses of the generation of renewable energy carriers (Heinzmann, Rudi, KIT-IIP)
16.30	Online: Sustainability and feasibility of the production of bio-oil integrated to Pulp Mill (Tero Joronen, BL2F coordinator, TAU)
17.00	Closing

Table 1: Schedule of the MTW

The presentations of the Summer School have been reviewed to remove sensitive data by the presenters. These have been published on the website (https://www.bl2f.eu/summerschool/) and are publicly available.

3.4 Social event

Appreciating the Finnish tradition of the coordinator and comparable to the first project meeting the social event at the evening of the first day of the summer school was held at lake Blausee in Altlußheim with catering and option to swim. A bus was booked to bring the attendees to the lake and to return them to Karlsruhe at 22.00. This informal gathering allowed participants to connect and create important networking opportunities for future biofuel research. Below are some impressions from that first evening gathering.

Figure 2: Impressions of the social event

4 Case studies

Prof. Dahmen gave in his introductory talk an overview on various routes to different intermediates for sustainable fuel applications, see slide 15 of his talk in figure 3. This was deepened in the talks of the different presenters, each focusing on individual intermediates.

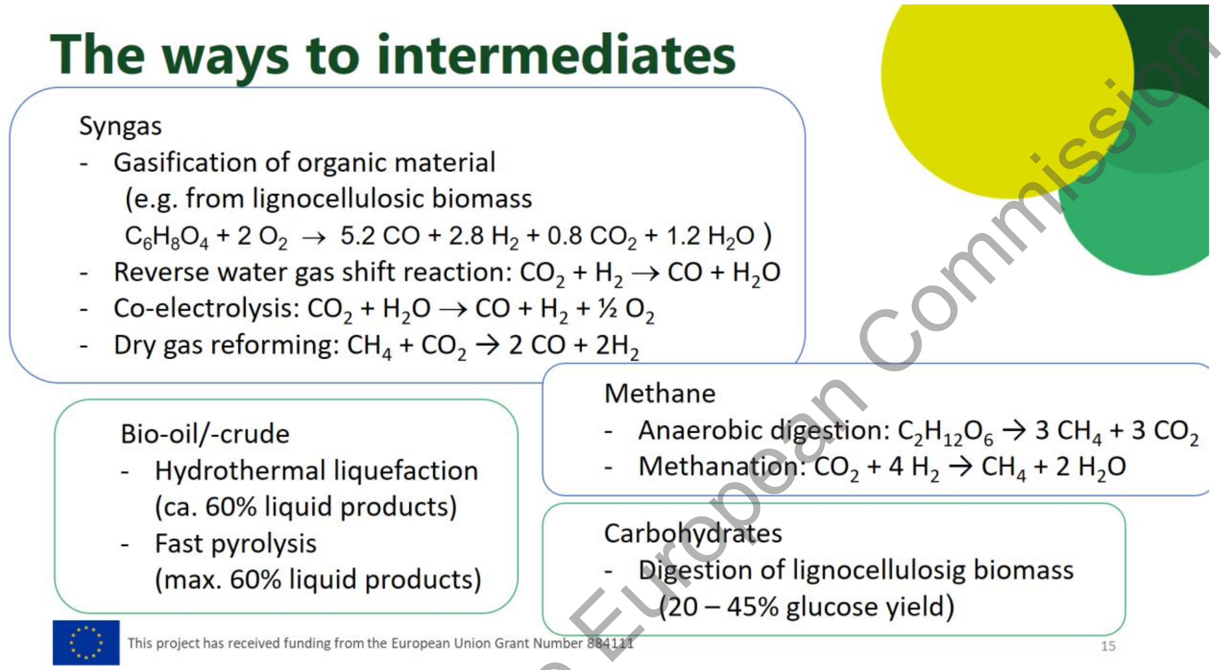


Figure 3: Overview of different intermediates, Prof Dahmen slide 15

The task for the participants was then to find a process example for one possible process chain utilizing the specific intermediate and to examine the following questions

- What does the process look like (schematic)?
- Which feedstocks can or are be used?
- Which products are obtained?
- Which fuel applications are possible?
- What is the fuel (energy) efficiency?
- What do you think are main advantages/disadvantages of the process?

Afterwards the results were presented in a short talk together with a simple process scheme.
4 Groups were realized working on the intermediates HTL biocrude, fast pyrolysis bio-oil, methanol and alcohol from lignocellulose. The following chapters on the case studies are the direct output of the student work written down by the moderators.

4.1 Group HTL Biocrude: Recovery of municipal waste via HTL (moderator M. Wörner)

- **Use of municipal waste (MSW)**
    - Heterogeneous collection of waste material
    - Around 1 -2 billion tons per year worldwide
    - Typical use: recycling, energy production, landfill storage
    - HTL can combine recycling or upcycling together with energy production even for waste ending up at landfills
- **First step: pre-treatment**
    - Mechanical/physical separation of metals and other inorganic compounds (possible recycling?)
    - Slurry preparation to achieve pumping feasibility
        - Milling
        - Mixing with water and additives
- **HTL of MSW slurry**
    - T = 300 – 400 °C, p = 250 – 350 bar
    - Four product phases: gas, biocrude, aqueous phase, char/solid residue
    - Gas goes through gas treatment, maybe recycle stream of burnable compounds (CH_4, H_2, …) for reactor heating
    - Possible char/solid applications: activation, fertilizer use (depending on composition), recycling of nutrients?
    - Aq. Phase contains organic compounds soluble in water and inorganic compounds; options are APR, recycling, extraction of organics for further processing in biocrude stream
    - Biocrude as main product
- **Hydrotreatment of biocrude**
    - HDO/HDS/HDN: removing oxygen, sulfur and nitrogen
    - Product can be processed in a refinery to produce fuels

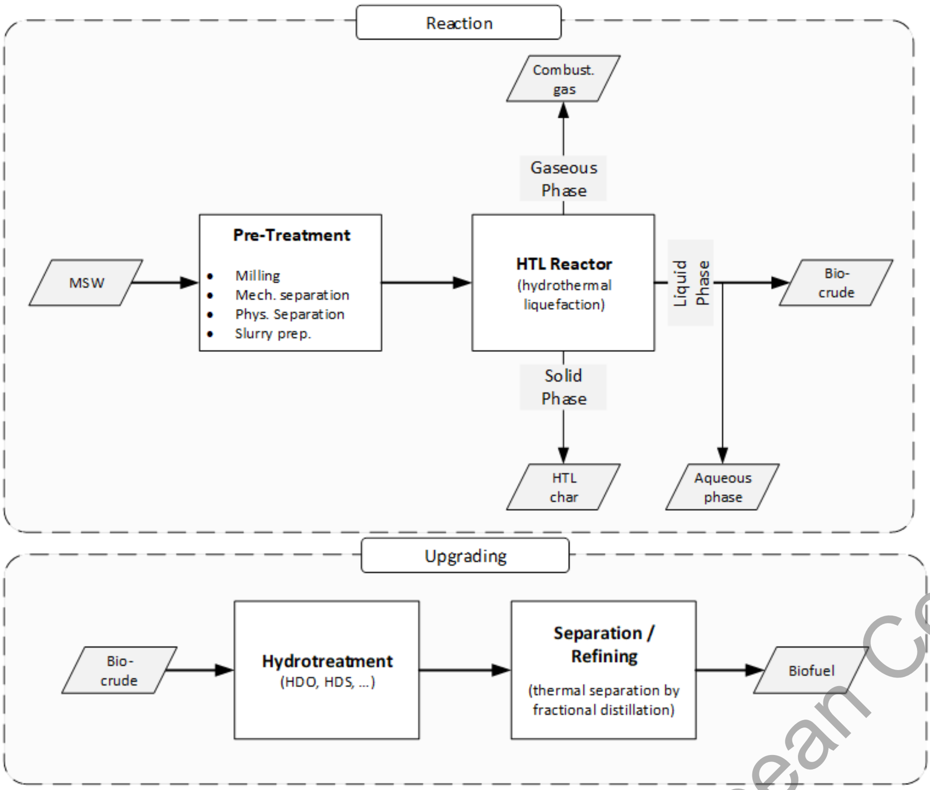


Figure 4: Process chain to MSW treatment via HTL

4.2 Group Fast pyrolysis bio-oil: Novel biorefinery on forestry residues (moderator F. Fonseca)

Forestry residues are common side products from the wood industry in several countries in Europe. Currently, they find several uses as sources of heat for combined heat and power or process heat or are shredded for the production of wood aggregate for construction and furniture A. Demirbaş, "Relationship between Initial Moisture Content and the Liquid Yield from Pyrolysis of Sawdust," *Energy Sources*, vol. 27, no. 9, pp. 823–830, 2005, doi: 10.1080/00908310490479042). The process presented in this work depicts an alternative valorization pathway for these residues for the production of jet fuel-, diesel- and gasoline-range products.

Fast pyrolysis is a thermochemical process in which a feedstock is subject to high temperature under an inert atmosphere, to crack macromolecules into a range of products. The process is typically operated at around 500 °C, with a low solid and gas residence time, to minimize the interactions between the gaseous products and the solid phase that lower the quality of the final products. The ash content and moisture content in the biomass are relevant control parameters and are preferably kept as low as possible.

The *bioliq®* concept contemplates the conversion of solid biomass residues to conventional fuels via gasification and a menthol-synthesis route. Solid biomasses, like forestry residues, present a very low energy density, substantially increasing these materials' transportation costs. Within the *bioliq®* concept, solid biomass is converted into an intermediate liquid product of high energy density and is more economical to transport. Conversion (fast pyrolysis) units are placed close to the biomass production centers, enabling the synthesis of fuels on a higher scale. The main products of these units are a pyrolysis gas, pyrolysis cokes, and two (or more) liquid phases, one of them named the bio-oil. (N. Dahmen, et al., "State of the art of the bioliq process for synthetic biofuels production," *Environ Prog Sustain Energy*, vol. 31, no. 2, pp. 176–181, Jul. 2012, doi: 10.1002/ep.10624.)

In comparison with other fast pyrolysis processes that traditionally employ fluidized bed systems, the one considered in the *bioliq®* materialization employs a twin-screw reactor, which enables the recovery of a high fraction of the cokes. Coke is a fine solid with a high carbon content that presents a high energetic potential but also presents possible uses as activated carbon, a carbon source for the industry and soils, or can be used as a carbon sink and incorporated into solid materials (A. Funke *et al.*, "Fast pyrolysis char - Assessment of alternative uses within the bioliq® concept," *Bioresour Technol*, vol. 200, pp. 905–913, Jan. 2016, doi: 10.1016/j.biortech.2015.11.012.). However, this process generates large amounts of an aqueous condensate phase that contains a substantial amount of oxygenate acid solutes, thus requiring costly wastewater treatment, but studies concerning its valorization as a fermentation substrate are undergoing.

The process presented by Shi et al. (W. Shi, et al., "One-Pot Conversion of Bio-oil to Diesel- and Jet-Fuel-Range Hydrocarbons in Supercritical Cyclohexane," *Ind Eng Chem Res*, vol. 53, no. 28, pp. 11557–11565, Jul. 2014, doi: 10.1021/ie501682r.) makes use of supercritical hydrodeoxygenation of the produced fast pyrolysis bio-oil to remove oxygen and increase the saturation of the final product in a single step. The studies have been conducted in lab scale (10 mL) and present promising results, due to a (reportedly) very high conversion (81%) of the highly oxygenated bio-oil (O = 26 wt.%) into an upgraded hydrocarbon mixture (O < 1 wt.%) of excellent quality. The upgraded mixture contains 90% of diesel- and jet-range hydrocarbons and 7% of gasoline-range and could be introduced into a conventional refinery process for separation and valorization. Supercritical processing is a mature technology, but no information was found about commercial catalyst conversion.

The biorefinery is partially presented in Figure 4. In it, forestry residues are dried to a low moisture content and fed into a twin-screw reactor where they suffer fast pyrolysis (500 °C). The subsequent aerosol mixture is run through a heated cyclone to remove the cokes, then through two or more fractional condensation steps to recover distinct liquid products. The fast pyrolysis process here employed is directly lifted from the setup employed at the current materialization of the *bioliq®* plant and is described in more detail in A. Niebel *et al.*, "Fast Pyrolysis of Wheat Straw—Improvements of Operational Stability in 10 Years of Bioliq Pilot Plant Operation," *Energy & Fuels*, vol. 35, no. 14, pp. 11333–11345, Jul. 2021, doi: 10.1021/acs.energyfuels.1c00851.).

The first liquid condensates, named the bio-oil, corresponding to around 30 wt.% of the original biomass and containing a water fraction of approximately 15 wt.%, are led into a supercritical HDO system, operating at 300 °C and 50 bar of H_2, using cyclohexane as the solvent medium, and Ni/ZrO_2 as a solid catalyst, as described by Shi et al., see above. It is important to keep in mind that the process described by Shi et al are water-free before hydrotreatment, as they make use of hydrothermal liquefaction bio-oil instead of fast pyrolysis bio-oil, therefore a water removal step may be paramount for the correct use of the process.

The coke fraction and the aqueous condensate, corresponding to 25 wt.% and 20 wt.% of the biomass are recovered as side products, while the pyrolysis gas is used to supply the reactor heat demand, as per (F. G. Fonseca, et al., "Moisture content as a design and operational parameter for fast pyrolysis," *J Anal Appl Pyrolysis*, vol. 139, pp. 73–86, May 2019, doi: 10.1016/j.jaap.2019.01.012.).

Based exclusively on the produced hydrocarbons, the process presents a carbon recovery of 35%, and an energy recovery of around 48%, based on the values in the original biomass.

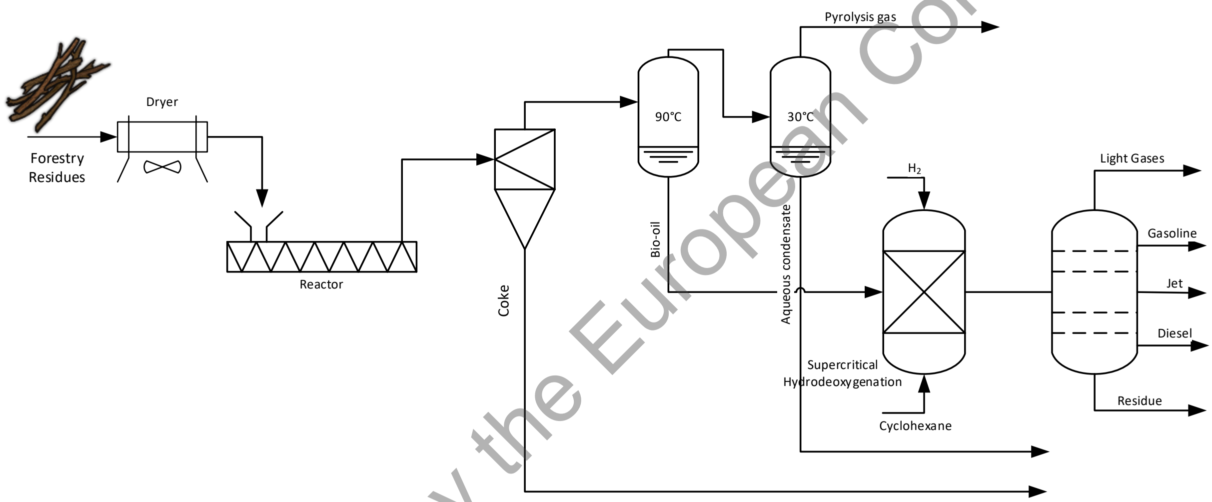


Figure 5: Fast pyrolysis treatment of sawdust

In this document, we presented a possible strategy for the production of conventional fuels from forestry residues by using fast pyrolysis and supercritical hydrotreatment. The process makes use of sawdust and forestry residues readily available in several European countries.

The process produces a mixture of hydrocarbons that can be easily integrated into the routine processing of a conventional petroleum refinery. The fast pyrolysis process is energetically self-sufficient and produces as a side product a coke which can be used as a fuel, as well as a carbon source or a form of carbon capture. The carbon source for this process is entirely renewable and does not compete with food or animal feed production like traditional biofuels do.

However, the process is in a low stage of maturity. While the individual processes may be commercial separately, these processes have not been scaled-up and properly tested in the configuration we proposed. Moreover, drying, water separation, wastewater treatment, reagent and solvent requirements, as well as catalytic supercritical hydrodeoxygenation add operational costs to the process and may further reduce the competitiveness of this biogenic fuel product.

4.3 Group alcohol: Lignocellulosic biomass to jetfuel alcohol (moderator H. Steinweg)

Input – C Source: No Sugar crop -> Woody Biomass to avoid tank vs. table issues
Which woody biomass? -> Sugar cane Bagasse as typical biomass residue in brasil / Sao Paolo region

Output – Product:
Main: Jet Fuel
Side: - Biomass pretreatment leftover – Filter cake
- Yeast cells
- Vinesse as broth distillation bottom product
- CxHy chains – Heavy fraction
- CxHy chains – Light fraction (recycled into reactor)

Steps:

1. Mechanical pretreatment: Size reduction from BM-as-delivered
2. Physical pretreatment: Steam explosion to open BM-structures for further treatment
3. Enzymatic pretreatment / hydrolysis: Degeneration of sugars to Mono-/Dimers
4. Separation: Filter press to remove residual solids
   ➔ Main USP intermediate: Liquid containing sugars, no solids
5. Reaction 1: Fermentative conversion sugars to ethanol with yeast, Batch fermentations in parallel
6. Separation: Filtration / Centrifugal separation of Yeast cells and debris
7. Purification: Distillation to separate Ethanol from broth, concentration increase from 5 % to 95 %
   ➔ Main USP product: 95% EtOH, sufficient purified for EtO synthesis
8. Reaction 2: Chemo-catalytic conversion of ethanol to olefins at elevated tempratures (300 – 500 °C) and moderate pressure, residence time and flow controlled according to C9-C14 Jet-Fuel spec.
9. Purification: Distillation to separate light, target and heavy fractions. Light fraction is recirculated into reactor 2. Target C9-C14 cut is target product.
   ➔ Main DSP product: Jet-Fuel in C9-C14 spec.

Applications of side product:

- Biomass pretreatment leftover – Filter cake
- Biomass pretreatment leftover / filter cake could be processed through HTL to achieve smaller and more homogenious molecular sizes by precipitation

and removal of anorganics, achieving another side stream to be processed towards value-added products. In simplified application this side product stream could deal energetic utilisation.

- Yeast cells

- Yeast is a valuable animal feedstock, providing nutrients and being tasteful, especially for omnivore / carnivore due to high concentrations in amino acids. If the process requires a GMO to digest the sugar varieties, an upfront inactivation step is required.

- Vinesse as broth distillation bottom product

- The bottom product of the Ethanol fermentation broth distillation is vinesse. Here, all non-volatile components are contained, both not converted educts, side products and non-separated cell debris – dissolved in water. Depending on the energetic value, biogas production to convert residual carbon into energetic utilisable methane might be an option.

- CxHy chains – Heavy fraction

- The heavy fraction of the EtO-synthesis, not meeting specs of Jet fuel grade, can be cracked to smaller chain lengths. Resulting stream can either be recirculated into the reactor or might meet another fuel grade.

- CxHy chains – Light fraction (recycled into reactor)

- The light fraction of the EtO synthesis is recirculatable into the EtO reactor, except a purge stream. The purge stream depletes all components with lower boiling point than target fraction, not participating in chain prolongation (Water, contaminants, …).

Conversion efficiency estimation:
The Conversion from sugar cane biomass to 95% EtOH is expected to achieve a 20 % (kg/kg) Yield, whereas half of carbon is expected to be converted from sugar cane bagasse into Ethanol.
The Conversion from 95% Ethanol to Jet-Fuel is expected to achieve a 50 % (kg/kg) Yield. Such chemo-catalytic processes utilise higher energetic, purified and uniform feedstocks, so a higher conversion efficiency can be achieved.
In general, the first conversion step should achieve an energetic concentration, whereas the second conversion step achieved a monetary upgrading from bulk chemical to target product. Combined Yield is expected at 10 % (kg/kg), whereas energetic efficiency from Feedstock utilised to Main product produced is expected at 24 % (kJ/kJ). Here, total massflow out and in are set in relation by their corresponding HHV ((0,1 [kg/kg]* 43 [MJ/kg]) / 18 [MJ/kg]).

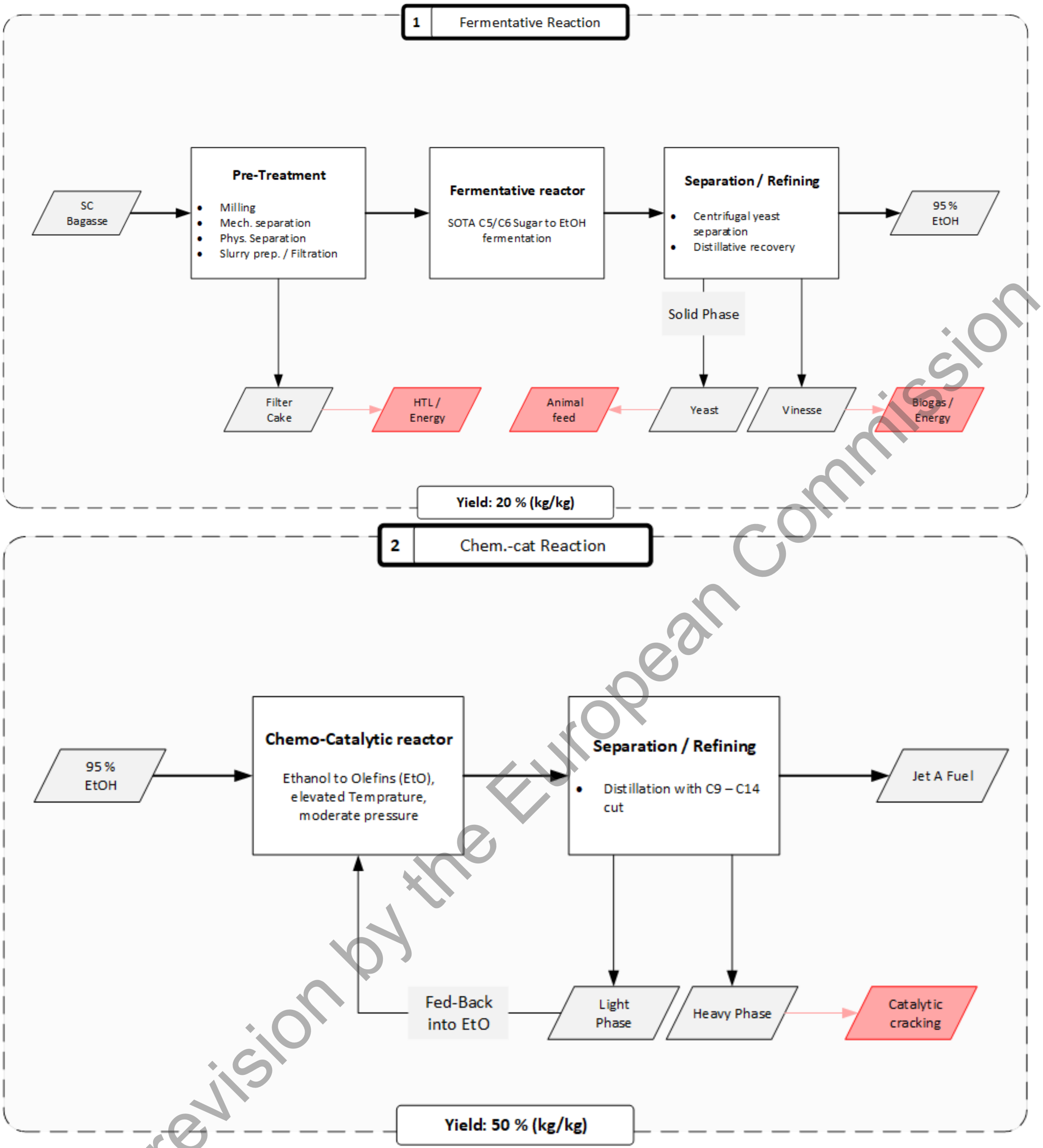


Figure 6: Treatment of bargasse

4.4 Group Methanol synthesis (moderator M. Herfet)

- What does the process look like (schematic)?

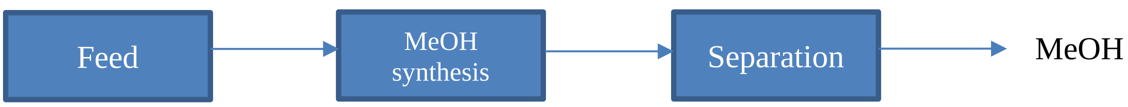


Figure 7: Methanol synthesis

- Which feedstocks can or are be used?
    - Syngas (biomass, CO_2 + H_2, CO + H_2, CO + CO_2 + H_2)
- Which products are obtained?
    - Methanol
- What is the fuel (energy) efficiency?
    - Exergy efficiency: 43.5% - 44.5%

5 Post-event statistics

5.1 Registration and Attendance

Considering the niched area of research by this scientific community, the results are especially positive , showing a high level of interest, illustrated by the registration/real attendees' ratio (see Table 3 for details on the total number of registrations and the number of participants for both day 1 and day 2). Furthermore, Figure 8 and Figure 9 offer insights into the affiliation of the participants and their respective nationalities. These statistics and data demonstrate the strong engagement and active participation of attendees, highlighting the significance and relevance of the topics discussed during the event.

Registrations total + presenters	Attendees Day 1
34	24
Presenters	**Attendees Day 2**
10	19

Table 2: Number of registrations and attendees

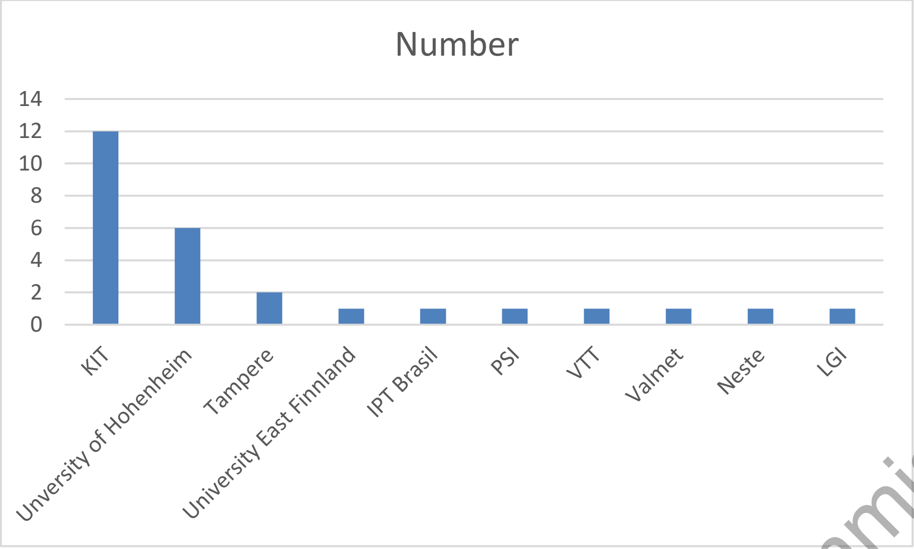


Figure 8: Affiliation of the participants and presenters

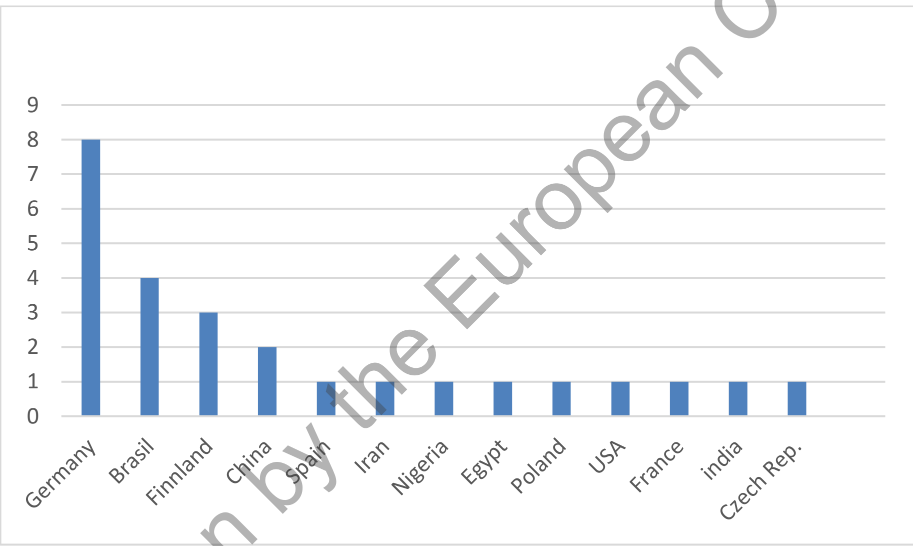


Figure 9: Nationality of the attendees and presenters

Figure 10 shows two group photos made on day 1 in front of the IKFT building and on day 2 during the lay tour at KIT in front of the Bioliq plant.

Figure 10: Group photo of day 1 in front if the KIT-IKFT and of day 2 in front of the bioliq plant during the Energy lab tour

5.2 Post-event survey

A survey was created to measure the satisfaction of the Summer school attendees. About 7 responses have been collected, the questions are included in Annex II.

The event was successful and the participants were satisfied; 4 rated satisfied and 3 very satisfied. They particularly appreciated the content of the presentations:

- *"Interesting topics"*
- *"Interesting presentations from the invited guests",*
- *"It's a great experience to learn about other people's research"*

- *"It was really a good opportunity to meet other researchers working in my research topic, share experiences between each other, and open new collaboration opportunities"*

One comment regrets that some of the invited speakers left earlier.

Asking how one could make use of the inputs or data presented, following comments were given

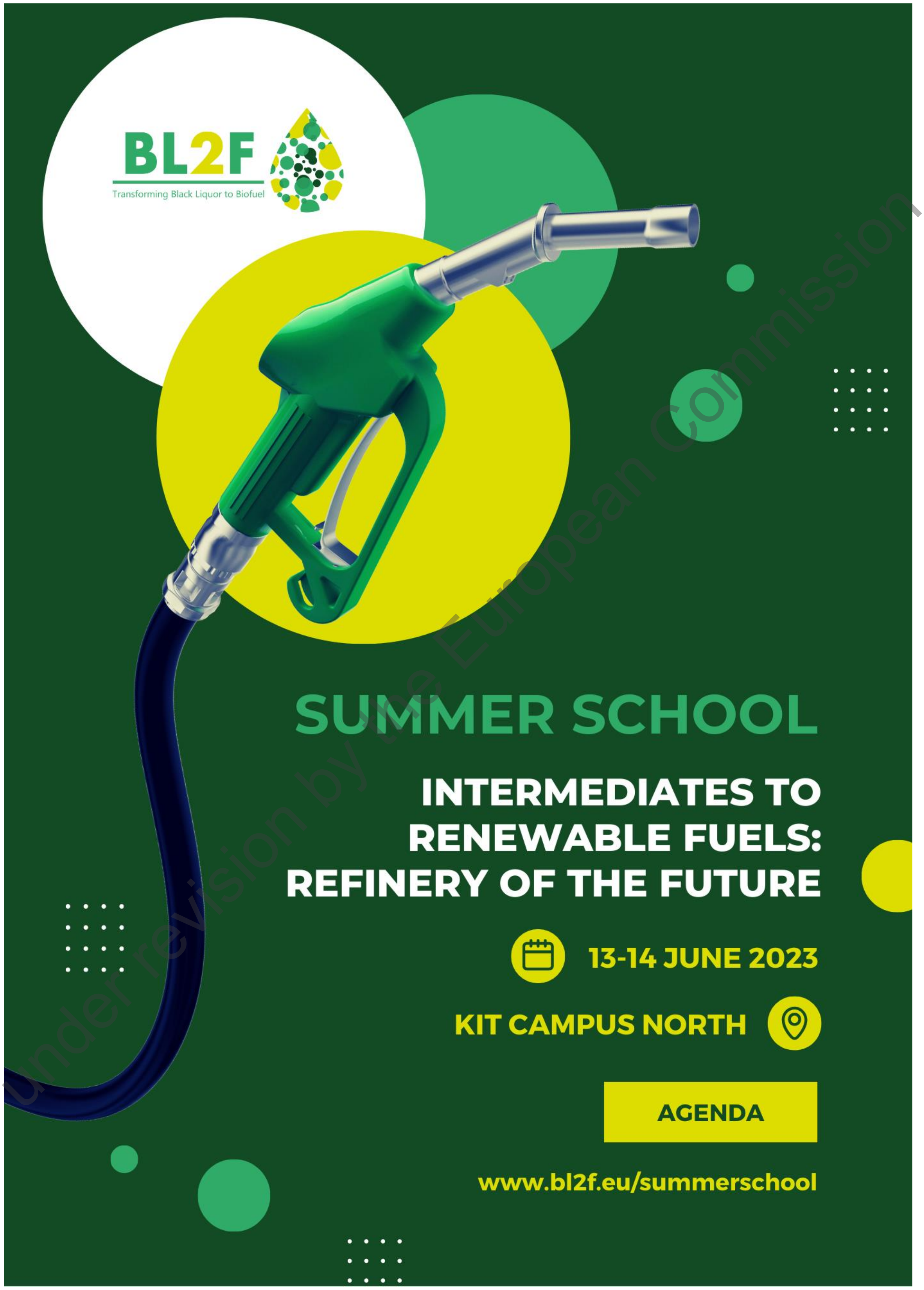
- *"It helps to Look over the Border of my own Research topic.",*
- *"Increasing knowledge of topic, possible employment paths",*
- *"It will be great to learn from the data presented during the summer school and I am waiting for it to learn more from these work experience.*

We also asked for further engagement with BL2F or the consortium after the Summer School and got the answer

- *"Yes, of course it would be a great experience "*

As a conclusion of the summer school we can say that the participants and students appreciated the event and agreed to become more aware of the idea, possibilities and potential of feedstock conversion to fuels and chemicals, via HTL and its downstream processing.
The summer school exchanges brought benefits to current researchers, allowing them to showcase their latest research to younger generations. It was also an opportunity for the researchers to incorporate the knowledge and perspectives from the future generations into their research. By doing so, we recognized that the participants are the next generation of leaders in this research field and would play a vital role in shaping what is to come.
This mutual exchange of ideas and insights enriched the learning experience for all participants and fostered a collaborative environment for advancing the field of study.

Annex I: Leaflet for the participants



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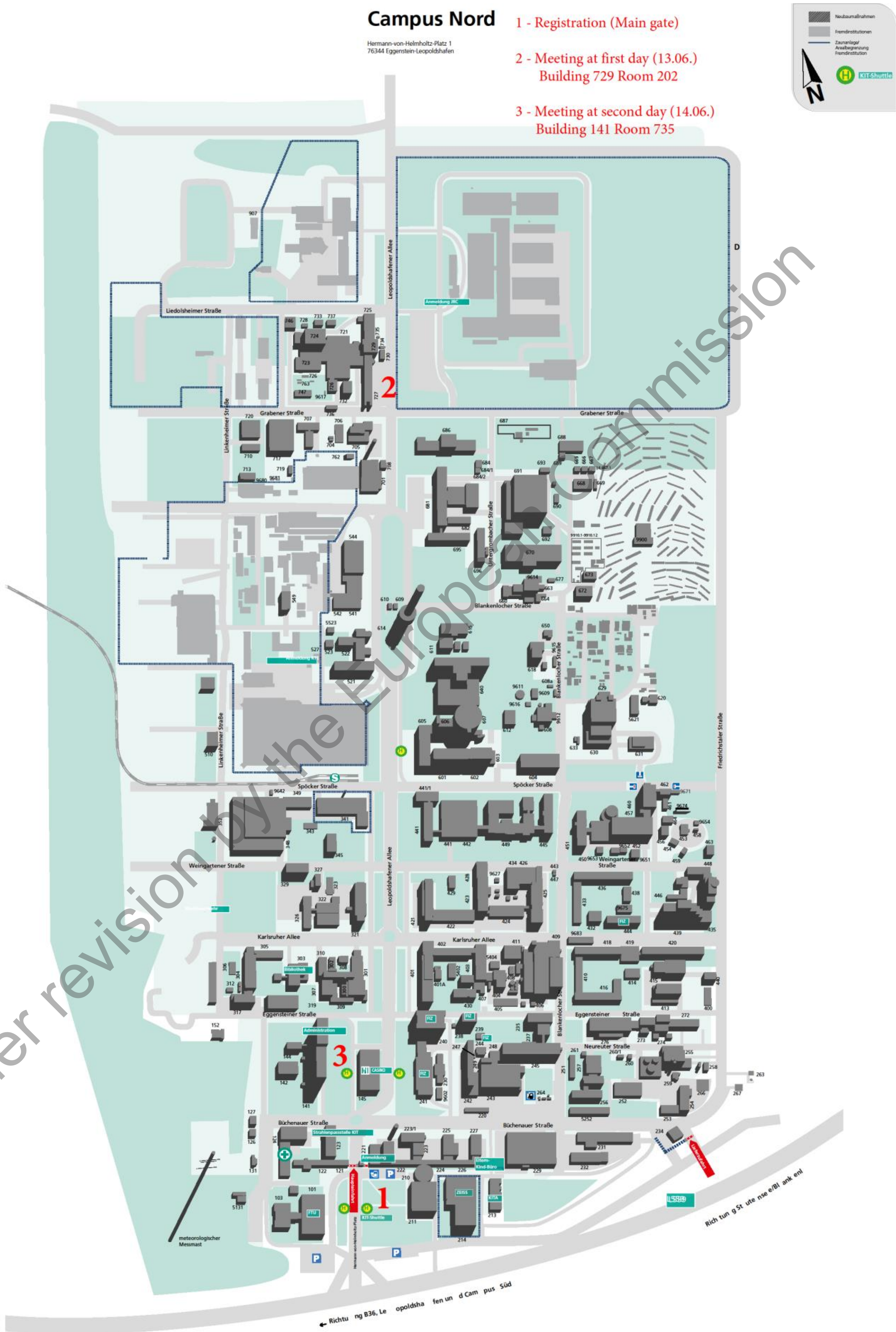

Campus Nord
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
1 - Registration (Main gate)
2 - Meeting at first day (13.06.)
Building 729 Room 202
3 - Meeting at second day (14.06.)
Building 141 Room 735
Liedolsheimer Straße
Leopoldshafener Allee
Grabener Straße
Linkenheimer Straße
Blankenlocher Straße
Spöcker Straße
Weingartener Straße
Karlsruher Allee
Eggensteiner Straße
Neureuter Straße
Büchenauer Straße
Friedrichstaler Straße
KIT-Shuttle
meteorologischer Messmast

Summer School - Evening event 13.06.2023

We invite you to our evening event at a lake, where we will serve a delicious paella. We want to spend with you and other guests a relaxing evening by and in the water, enjoying Spanish cuisine.
Paella is a traditional Spanish dish made with rice, vegetables, meat or seafood and is known for its unique taste.
We look forward to spending a wonderful evening with you!

Survey - BL2F Summer School

Annex II

Post-event survey - BL2F Summer School

Thank you for attending the BL2F Summer school: *Intermediates to renewable fuels: Refinery of the future.* Please fill out this short, anonymous post-event survey. We hope to see you again soon!

1. How did you hear about the summer school?

- Somebody told me about it
- BL2F social media accounts
- Other social media accounts
- BL2F website
- Email invite
- Other

2. How satisfied are you with the content of the Summer School?

- Very satisfied
- Somewhat satisfied
- Neither satisfies or dissatified
- Somewhat dissatisfied
- Very dissatisfied

3. Please specify

Free text

4. How could you make use of the inputs or data presented?

Free text

5. Do you intend to seek further engagement with BL2F or the consortium after the event?

Free text