STSE report

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1.Work location

The short-term staff exchanges took place at the Karlsruher Institut fuer Technologie in Karlsruhe Germany, namely in Lab 03 “High Pressure Laboratory” in Karlsruhe Institute of Technology, locate in Engler-Bunte-Institute/Fuel Technology, in Engler-Bunte-Ring 1, from the 7th to the 18th of November. The work was planned in detail and supervised by Prof. Dr. Reinhard Rauch, with collaboration PhD Student Philipp Graefe, both from KIT's chemistry department.

2. Main goal

During this period, there were performed the calibration of the gas chromatograph through different gas mixtures, maintenance, modification, and operation of the Fischer-Tropsch reactor and as supplementary work, research, and summary of European projects (Horizon Europe) for future partnerships between KIT and IPP were investigated.

3. Activities

3.1. Laboratory and main experiment

To start activities, the PhD. Student Philipp Graefe performed an introduction to laboratory safety, as well as all the procedures to be followed in a work context. Then, I had the opportunity to work with the help of Master Student Valentine Honold, in the calibration of the gas chromatograph (GC) Shimandzu Nexis GC-2030. The GC was used to detect specific volatile elements in a gas phase mixture, to quantify the concentration of specific chemicals, namely nitrogen, argon, hydrogen, carbon dioxide and carbon monoxide. As with any form of quantitative analysis, proper calibration is the key to success in gas chromatography workflows, particularly in processes such as Fischer-Tropsch, where it will be necessary to know the mass balance in detail. For this reason, several analyses were carried out with different gas mixtures, until chromatograms were obtained (figure 1.) with an error of less than 1%.

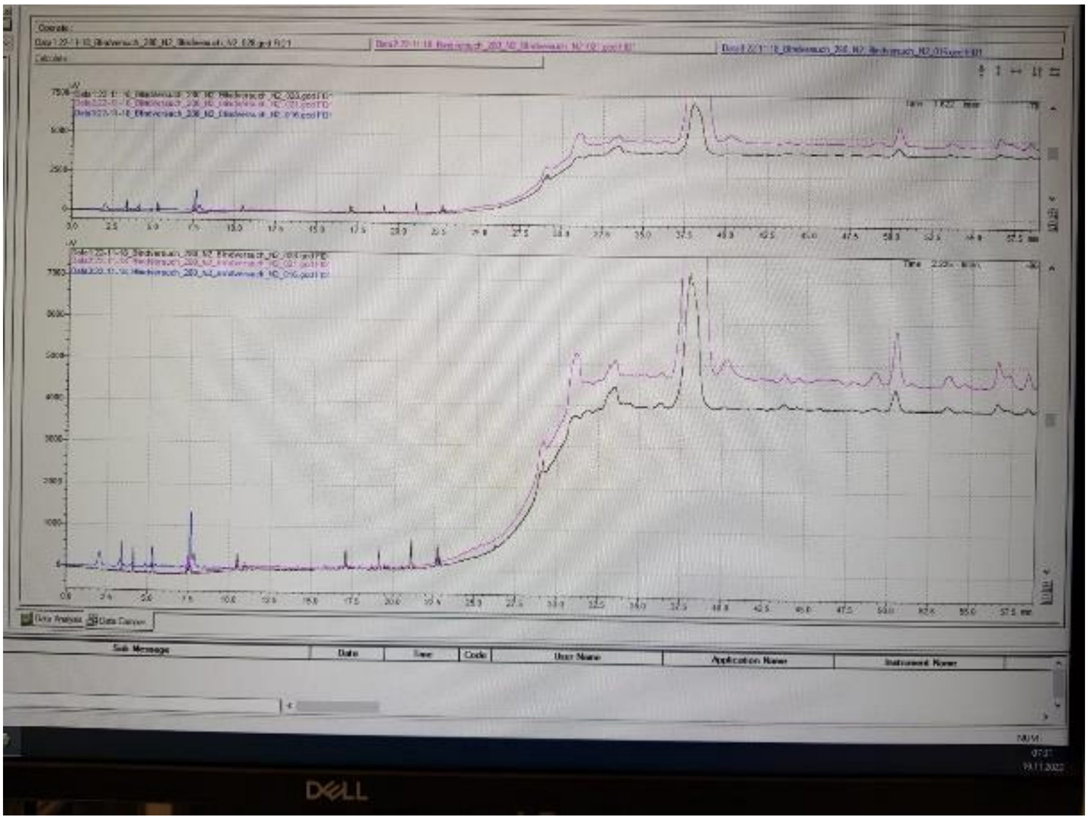

Figure 1. Comparison between chromatograms.

Evidently, the GC is a sensitive piece of equipment and needs to be studied. Although some analyses revealed small errors, it was necessary to test several columns to minimize the analysis errors (Figure 2.).

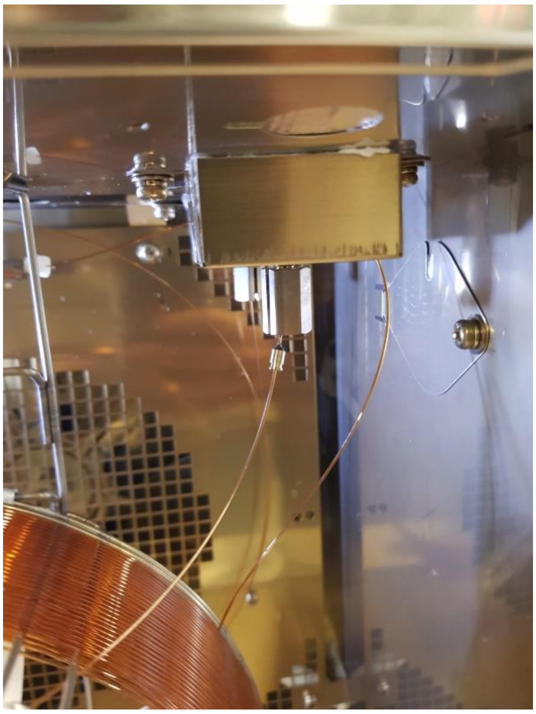

Figure 2. Installing new GC column.

To start work on the FTS reactor, it was proposed to study the equipment responsible for cooling/condensing the process products (huber minichiller 600), not only the mode of operation (temperature of 12 ºC), but also the different mixtures of coolant liquids that could be used in it, to avoid corrosion and a cooling performance (Figure 3).

Figure 3. Huber minichiller 600 regulations.

Subsequently, tests were started with the FTS reactor. All parameters were studied and checked, from pressures to temperatures. In this first start-up of the unit, a leak in the hydrogen line was detect. As a highly instrumented reactor, it was easy to detect the location of the leak, however, it made impossible to carry out tests and all efforts were channelled towards repairing the leak (figure 4). For the repair, it was necessary to remove all the thermal insulation, to reach the location of the leak and tighten the line connection well.

Figure 4.Hydrogen line leak repair.

After repairing the line, blank tests are started, just testing the entry of different gas mixtures, pressures, and operating temperatures. The gases were analysed through the GC and controlled by another analyser, named the ABB AO2020. Using the Mettler Toledo GPro500 equipment, the pressure was controlled, and the temperature was controlled through thermocouples connected to the control part of the reactor. At this point, it was necessary to carry out a new battery of tests with the GC, since the reactor outlet pressure (atmospheric) is different from the calibration bottle outlet pressure, which led to a new study of gas cleaning times in the line of analysis (Figure 5.).

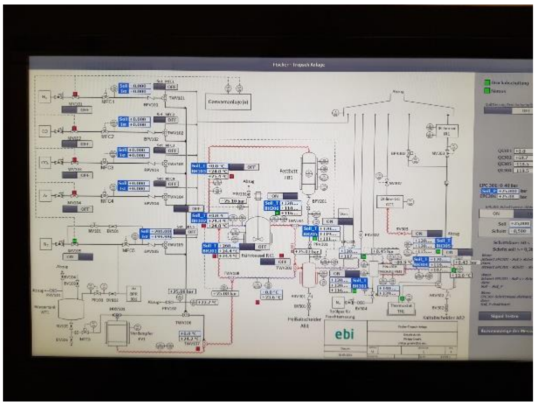

Figure 5. Cleaning the lines with nitrogen.

After several blank tests, it was discovered that the upper part of the stirrer, which had the control instrument, the temperature became high as the test progressed. So that the agitator controls were not damaged, it was placed laterally, thus using a belt that worked through toothed wheels (Figure 6.). After the test, it was also verified that there was a gap where the agitator shaft bearings are located (Figure 7.), which was repaired. However, this system did not become viable, due to the high speed of agitation and trepidation of the same, causing the belt to jump.

Figure 6. Stirrer using a belt that worked through toothed wheels.

Figure 7. Ball bearing repair.

After maintaining the equipment and the cleaning of the gas line, the first tests were carried out with gases only, that is, without the addition of catalyst and squalene. The conditions were a temperature of 250 ºC in the reactor, 25 bar in the reactor and in the low-pressure zone about 0.5 bar, stirring at about 800 rpm and a gas mixture of 11.111% for argon, 22.222% for carbon dioxide and 66.666% for hydrogen (Figure 8.).

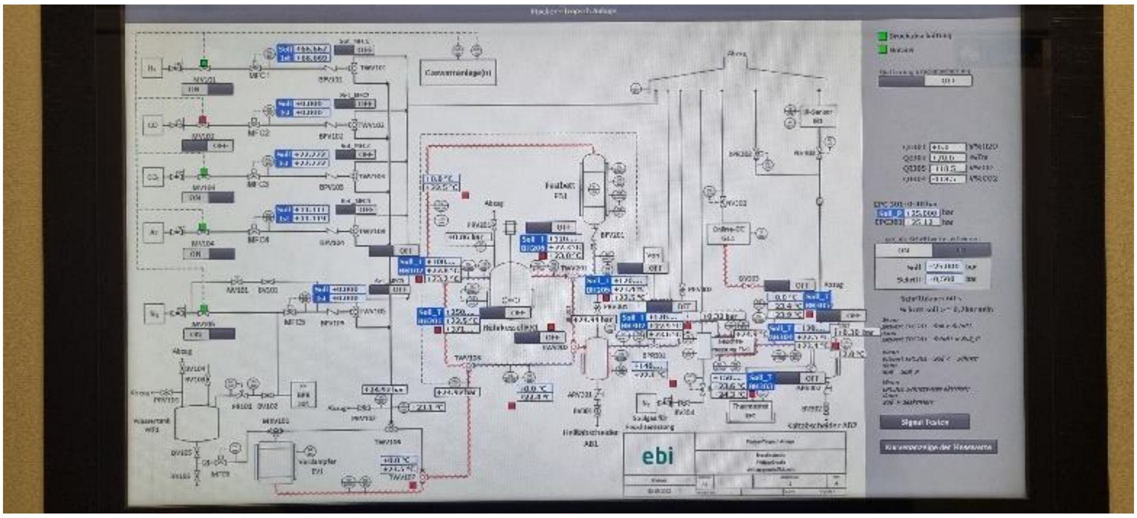

Figure 8. First experiment – controls.

The tests proceeded as expected, with no anomalies occurring in the GC analyzes or with the mechanical and electrical parts of the reactor. After the experimental procedure, the reactor was opened and cleaned.

Then, tests were carried out with the addition of the catalyst, 10g (Fe-K-Cu/Al_2O_3), and 300ml of squalene ($C_{30}H_{62}$), at the same temperatures and pressures conditions of the blank tests.

Unfortunately, time passed quickly, and there was no opportunity to carry out the analysis related to the liquid and gaseous fraction of the tests.

3.2. Open calls

As supplementary work, it was proposed to examine new calls from Horizon Europe, where a partnership between KIT and IPP could apply. In this sense, a small summary of the most appealing calls for a collaboration was made, which were the following:

- HORIZON-CL5-2024-D3-02-03: Development of smart concepts of integrated energy driven bio-refineries for co-production of advanced biofuels, bio-chemicals and biomaterials;
- HORIZON-CL5-2024-D3-01-10: Next generation of renewable energy technologies;

- HORIZON-CL5-2023-D3-03-02: Integration of renewable gases, other than hydrogen or methane, and which have not access to gas grids and interfacing with electricity and heat sectors;
- HORIZON-CL5-2023-D3-02-07: Development of next generation advanced biofuel technologies;
- HORIZON-CL5-2023-D3-02-01: Development of near zero-emission biomass heat and/or CHP including carbon capture;
- HORIZON-CL5-2023-D3-01-06: Demonstration of advanced biofuel technologies for aviation and/or shipping.

Subsequently, a meeting with the professor and Philipp was held to better address the issue. Other types of projects were also discussed at the meeting, such as bilateral projects, as well as future partnerships in terms of student exchange.

4. Final Considerations

The contribution of short-term staff exchanges to my training was a great asset.
I had the unique opportunity to work with a Fischer-Tropsch reactor and its analysis processes at the Karlsruher Institut fuer Technologie, despite the theoretical knowledge I had about the process, the knowledge acquired was a great asset to my training.
I feel privileged to have helped and performed FTS tests and analysis of the gas phase of the process.
And last but not least, a special thanks to the KIT team, it was a huge honour to share this whole experience with Prof. Dr. Reinhard Rauch, PhD Student Philipp Graefe and Master Student Valentine Honold, who were always available and very cooperative. Also, a special thanks to Prof. Dr. Paulo Brito and co-workers from IPP for their help and availability.
These aspects are fundamental in the labour market, since what is sought is people with experience in new technologies and who can quickly integrate into a work group, fostering team spirit.