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V1.0	09/01/2024	ALPES	First complete version
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Executive summary

This is a publishable final activity report of HYDROPTICS, covering main aspects of the work, objectives, results, and conclusions, including the publishable results of the final plan for using and disseminating the knowledge. This document only presents information that are openly accessible and public and will not delve into confidential or sensitive details.

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1 Introduction

This report describes the work, objectives, and results that were part of the HYDROPTICS project, followed by the list of project's objectives, work packages and a detailed description of the individual results stemming from said work packages.

2 Work packages list and relevant project results

WP1: Project Management

- Public Final Activity Report (this document)

WP2: Hydroptics Ethical, Legal & Regulatory aspects

- Public Reports on ethics, gender issues, GDPR, legal aspects, regulatory & standardisation activities.

WP3: Hydroptics requirements, use case definition and design of pilot services

- Preparatory activities

WP4: Quantum Cascade combs development and control electronics

- Novel Laser sources
- Sensing components and methodologies

WP5: Ancillary sensing components & methods development

- Balanced detection and interferometry using QCL sources
- UV-Vis-NIR Hyperspectral imaging components
- Sample conditioning for online sensing

WP6: Signal acquisition, processing & industrial process optimization algorithms

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WP9: Communication, Dissemination, and Exploitation of Hydroptics results

- Dissemination plan and material (website, logo, etc.)
- Reporting on the implementation of dissemination & communication activities: First & Final
- Report on IPR activities: First & Final
- Communication activities: First & Final

3 Description of delivered publishable results

3.1 Preparatory activities

The use of a frequency comb spectrometer combined with the information gained by a dual-DFB approach gives a high amount of sensitivity in a defined small wavenumber region. An important step of the HYDROPTICS developments included the study of oil products using mid-IR spectroscopy-based tools in the lab. The aim of this study was to pave the way for the development of the HYDROPTICS sensors. A broad range of different model substances were analysed, and the characteristic absorptions of the relevant vibration modes were assigned. results on the spectral properties of the targeted analytes guided the development in the right direction. A database was used to define characteristic absorption regions for the frequency comb spectrometer being developed in the HYDROPTICS project. Moreover, optimization strategies investigated have been employed to establish validation approaches for test facilities. Several plant critical process parameters have been explored. Consequently, an examination of the layout of these facilities has been conducted to identify the most effective strategy for validating the optimization algorithms. A model which predicts events in a real-life test facility has been developed. With the help of sensitivity analysis, certain optimization potentials have been identified. These optimization strategies can be validated by application of certain parameter changes at the test facilities and thorough measurement routines in combination of the developed sensor units.

Finally, the preparatory activities of the project include the development of a detailed overview of how the Hydroptics platform will be developed and lists the properties of the components that will be installed in the prototypes. The specifications and requirements regarded both the individual components, as well the whole HYDROPTICS modular platform and integration strategy.

3.2 Novel laser sources

Dual-comb and dual-DFB spectroscopies have been identified to be the most efficient technologies for HYDROPTICS project. These spectroscopic methods are possible thanks to the development of highly coherent mid-IR light sources, that are the QCLs. Among the main objectives of the project was to deliver novel advanced laser sources in the mid-IR, namely Quantum Cascade Laser (QCL) frequency comb and Distributed Feedback (DFB) QCL tailored for the HYDROPTICS needs in terms of specs and design criteria. The project achieved the successful fabrication of newly designed active region QCL devices, with the dedicated etching design for possibility of integration to a germanium on silicon beam combiner chip. The latter aimed to deliver a second wavelength QCL frequency comb generation that is necessary for aromatic hydrocarbon detection. The developed open a path towards fabrication of highly efficient, fully integrated, dual-comb and dual-DFB devices.

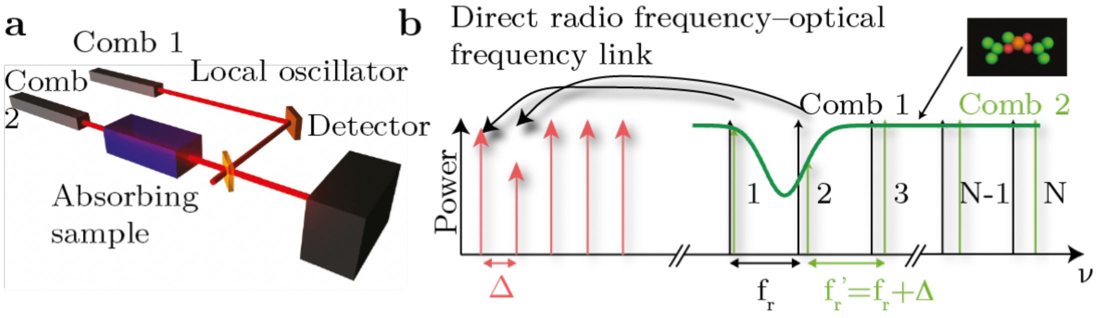

Figure 1: Principles of dual-comb spectroscopy

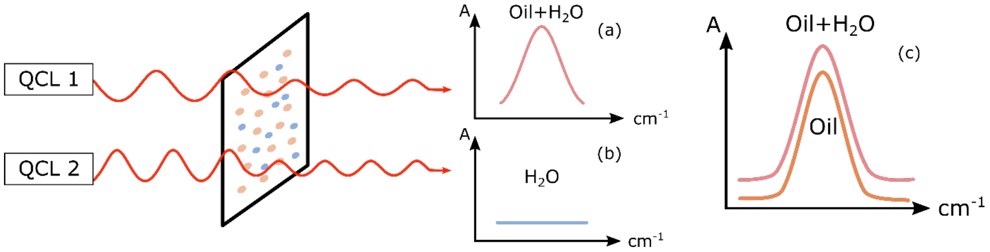

Figure 2: Drift Compensation measurement principle using dual-DFB QCL sources.

The design criteria, the fabrication, the post fabrication processes (etching, HR coating, cleaving) of the laser chips were addressed as well as a dedicated custom designed processing step has been performed for the possibility of integration of the laser devices to the germanium (Ge) on silicon beam combiner chip via the implementation of a new process. Also, a dedicated RF section has been designed and fabricated for QCL frequency comb devices, aiming to achieve better control and monitor the comb operation. These developments were crucial for the subsequent steps of the project, i.e., the fully integrated and stabilized dual-comb and dual-DFB spectrometer setup.

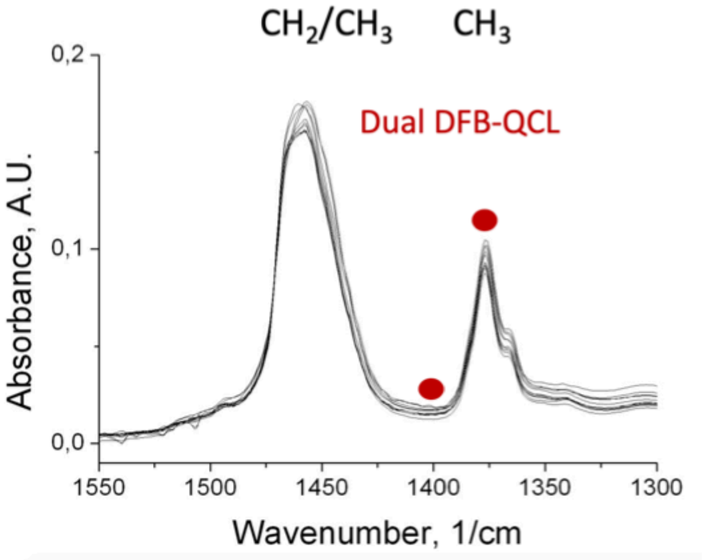

Figure 3. Absorption spectrum of oil. Red dots indicate the selected wavenumbers for two QCL-DFB lasers to continuously measure the oil content.

The DFBs developed by ALPES delivered output in the 18 mW range and were fully characterized.

The beam combiner relies on a Ge-on-Si integrated optical waveguide circuit, where a miniature beam splitter can be implemented using wafer-scale technology. The co-integration of the two III-V frequency comb chips was developed by IMEC by utilizing a 3D self-aligning strategy.

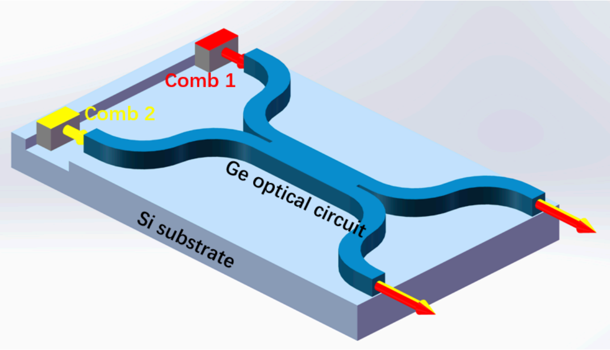

Figure 4. Sketch of the integrated beam combiner with the optical combs directly coupled to Ge-on-Si waveguides.

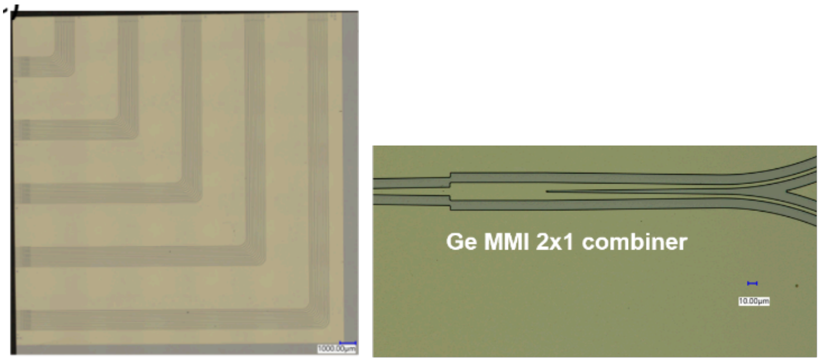

Figure 5. Microscope images of fabricated Ge-on-Si chips

Flip-chip bonding of QCLs on PIC is required to have an accuracy in the micron range and it was required to determine the bonding accuracy. The bond accuracy would be measured by powering-up the QCL and then analysing its output signal through the Ge waveguide. To be able to flip-chip mount the QCL devices on a silicon chip with Ge beam combiner waveguide, and to be perfectly aligned to the beam combiner waveguide entrance, a custom process design has been developed in close collaboration with partners IRsweep and IMEC. The approach includes both a vertical and a horizontal alignment. Despite the challenge, design and fabrication of Ge-on-Si beam combiner circuits were successfully completed and self-alignment flip-chip bonding to integrate QCL on the PICs was successfully demonstrated, marking a significant milestone in expansion of mid-infrared silicon photonics active components.

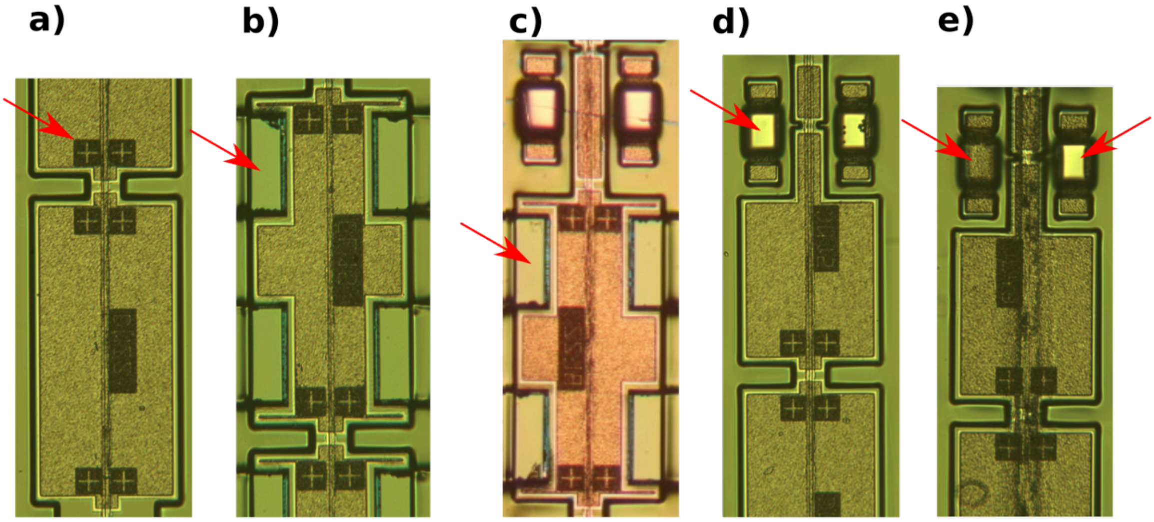

Figure 6. Photograph of several versions of the processed QCLs combs and DFB devices developed during the project.

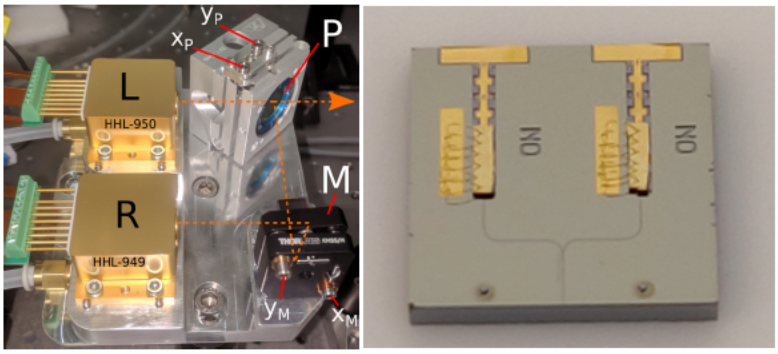

Figure 7. "Bulk" beam-combiner already reported in D4.6 (left); example of dual-wavelength DFB-QCL source based on a beam combining chip.

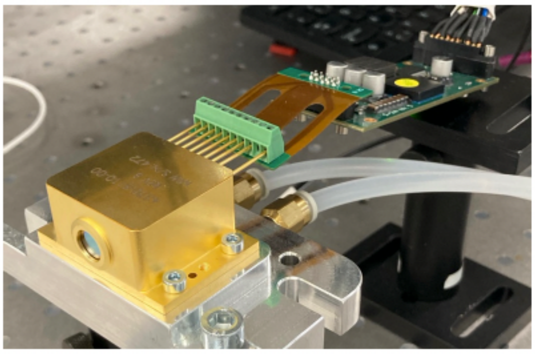

Figure 8. Fully packaged PIC in a HHL module connected to a S2-M driver.

Moreover, a strategy implemented by HYDROPTICS aimed to stabilize the spacing between all the lines of a QCL frequency comb using RF injection. A dedicated RF section has been designed and processed on the Fabry-Perot devices. RF sections for QCL frequency comb for efficient intermodal beat note signal extraction and manipulation allows more efficient RF injection locking of the comb devices.

Further to the laser chips, novel QCLs frequency comb drive and locking electronics were developed within HYDROPTICS. A specially designed layout of the dual laser drive was developed aiming easy integration in the spectrometer. In a second step, an entirely new control electronic was designed together with a new spectrometer while also new control software and firmware were developed to achieve the goal of a more integrated and cheaper overall system.

3.3 Sensing components and methodologies

The primary scope of HYDROPTICS was to develop tailored sensing solutions for the oil industry. The following sensing components and methodologies have been developed.

3.3.1 Balanced detection and Mach Zehnder interferometry using the dual DFB-QCL source

Inherent properties of QCLs such as their coherence or their polarisation enable the development of sensing schemes that go beyond classical absorption spectroscopy based on Beer´s law. Dispersion spectroscopy relies on the coherent nature of QCL sources and with the use of interferometry it allows for reliable liquid phase analysis by means of sensitive refractive index changes (dispersion) measurements. Polarimetric attenuated total reflection spectroscopy makes use of the fact that parallel and perpendicular polarized light shows different effective depth of penetrations. Hydroptics explored novel sensing possibilities with the aim to enhance the on-line sensing capabilities for oil-in-water measurements considering the use of extraction solvent as well as to aim for increased long-term stability, sensitivity, and selectivity in evanescent wave spectroscopy.

A dual DFB-QCLs based balance transmission Mach-Zehnder Interferometry (MZI) setup has been developed and validated by TUW. The absorption measurements performed with the balanced MZI setup demonstrated the advantageous capabilities using a dual DFB-QCL laser, with the calibration not being influenced by wet solvent which, in contrast, poses to be a big problem for a single DFB-QCL laser setup due to a baseline shift caused by trace amounts of water. This is a significant improvement when compared to the current state-of-the-art where a single DFB-QCL laser setup is used whose performance is adversely influenced by changing background absorbance of the background solvent.

Moreover, a polarimetric balanced detection setup has been developed. In this setup, the laser itself emits horizontally polarized light, the beam hits the entrance facet of the ATR element. By employing the novel HYDROPTICS sensing configuration, a significant improvement in long-term stability could be achieved. This scheme can be exploited for the development of a sensing scheme which makes it possible to quantify small amounts of phosphate in process water, we utilised the widespread concept of solid-phase extraction, which is normally used in analytical chemistry for the routine sample preparation and purification. Applied to ATR spectroscopy, this concept can be realised by applying a novel thin mesoporous layer with high affinity to the analyte to an ATR prism.

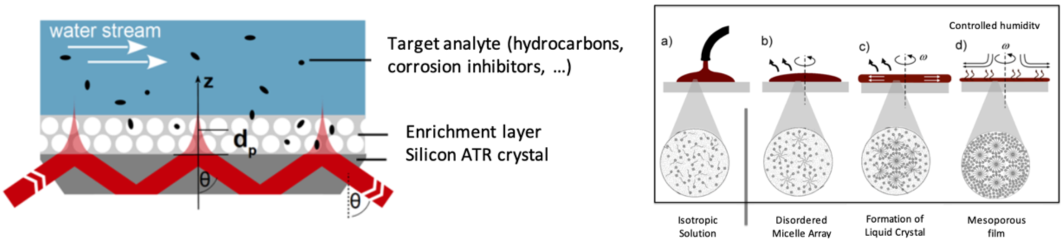

Figure 9: Left: ATR sensor concept employing an enrichment layer. Right: Schematic of the individual steps for producing mesoporous layers by spin-coating.

3.3.2 UV-Vis-NIR Hyperspectral imaging components

A UV-VIS-NIR Hyperspectral imaging components required for online sensing with the HYDROPTICS platform has been developed. A hyperspectral imaging module and the fluorescence imaging module were assembled to demonstrate the precise particle measurements. For the analysis of the solid particles a colour imaging system and a hyperspectral scanning system were built up. For the detection and size determination of the oil droplets a fluorescence imaging system was built up. Using the highly efficient sensing approach employed, most targeted particles could be detected with the hyperspectral setup with reflected light illumination, with the rest, as well as for the detection of oil droplets in water being detected by the fluorescence setup.

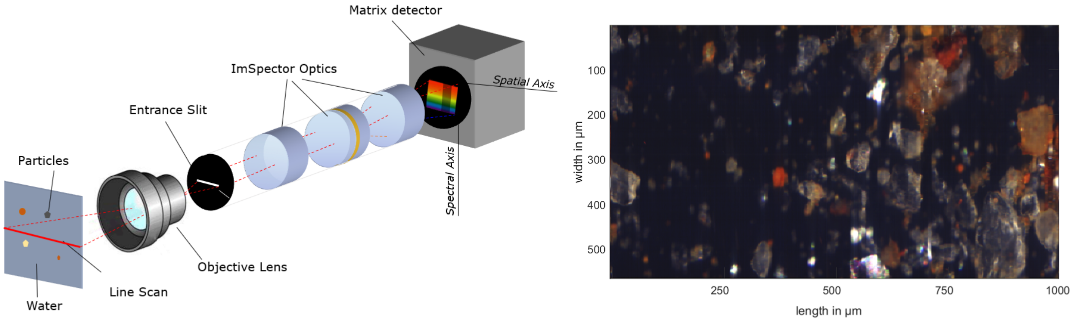

Figure 10: Scheme of the hyperspectral imaging system (left); RGB color image of particles in water using the HSI.

3.4 Sample conditioning for online sensing

Further to the development to the core photonics-based sensing schemes, novel sample conditioning for online sensing modules has been developed and demonstrated. An ultrasound assisted flow cell was built for the Hyperspectral Imaging module and the Fluorescence spectrometer to enable precise particle measurements.

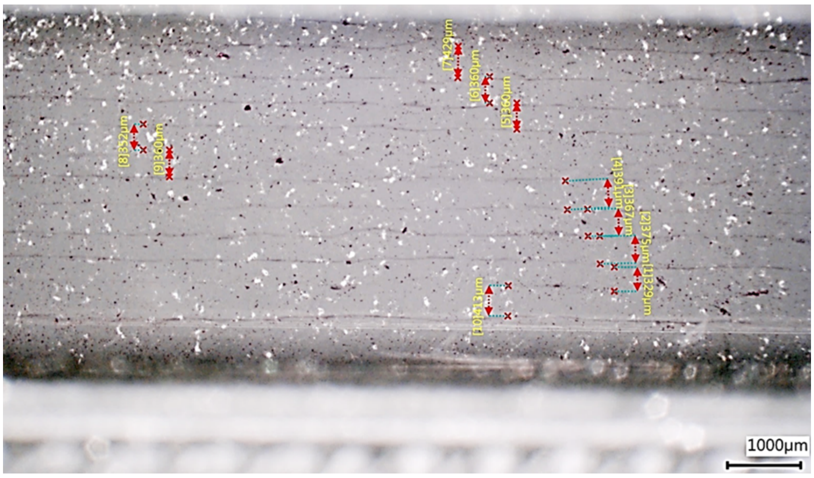

Figure 11: Acoustofluidic (ultrasound) cell with guided 10 µm polystyrene beads.

Moreover, an automated liquid/liquid-extraction module was assembled to extract the dissolved oil and to measure it with infrared spectroscopy. This extraction module is equipped with a novel centrifugal separator, which is necessary to separate the mixture of aqueous sample and organic solvent.

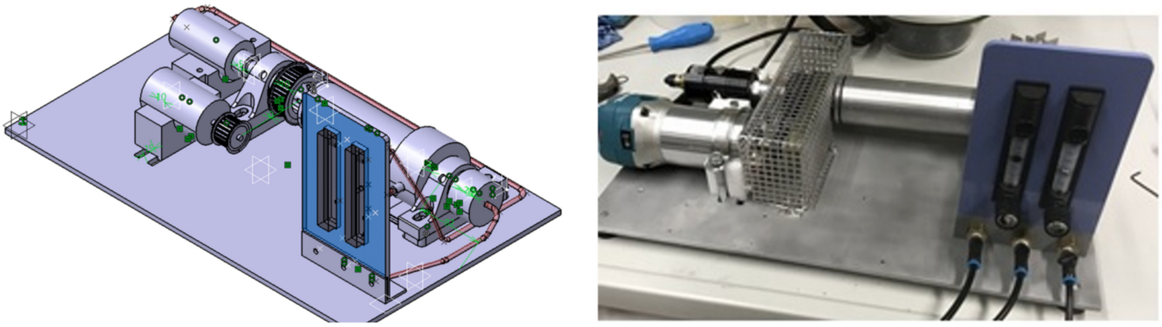

Figure 12: Centrifugal separator first prototype: 3D CAD Model (left); Lab model (right).

3.5 Digital twin of an exemplary water treatment process in the oil & gas industry

A digital twin was developed for an exemplary water treatment process in the oil & gas industry, using process simulation software and CFD to model the plant components in various scenarios. Flowsheet modelling and different calculation tools were applied to examine the physical parameters and effects in each unit. The CFD approach was based on open-source software, which provided a detailed view of the effects. The resulting digital twin was an improved virtual replica of the plant, utilising both approaches to allow for a detailed overview of all operational areas.

3.6 Sensor parameters of the model process

An area of interest selection algorithm that could reduce the HSI signal, considering both spatial and spectral information, was implemented successfully. The dimensionality reduction process was realised through a novel tensor based autoencoder framework. This process could provide results close to the state of the art, but could be trained using way less samples, and did not impose significant computational overheads, as it could be trained without needing any additional label information or learning paradigm additional to the autoencoder. The quality of the process could only be measured using annotated datasets. For this purpose, the performance was assessed using state of the art benchmarking datasets, specifically two datasets, and not in data derived from a project. The RFE method reduced the input signal in a way that the performance of supervised learning model was not affected. This was not the case with the method proposed here, which only needed a few data to work, without any extra information or models.

For the fusion of the signal with other parameters, a deep multi-channel architecture was created. The model used the simplified signal and other parameters as inputs and produced an output that could be shown. The goal of this was to create a model that could change the raw signal into a meaningful representation. Moreover, the model would allow testing the effect of other parameters in the quality of this process.

3.7 Modelling of CQAs based on CPPs

The quality of the water output was dependent on the critical process parameters (CPPs), which were identified by different methods. The gravity settling theory and the population balance equations (PBE) in computational fluid dynamics (CFD) software were used to analyse the oil residuals and the oil dispersion in the water. The models were able to simulate the oil emulsions in water and predict the separator efficiency and the emulsion characteristics.

3.8 Process optimization on an engineering level

Optimization strategies for oil and water separation plants were explored based on the digital twinning and the CFD modelling approaches. The effects of the overall plant were analysed using the process simulation model (Digital Twin), which could produce dynamic effects in the plant components. The 3-phase separator and the flotation cells were identified as critical units and examined by CFD simulation. Based on these simulations, suggestions were given for process optimization of the plant. A macro-scale simulation model of the entire plant was created, which could optimize the monitoring and the design of the plant components. A micro-scale simulation method was used to study the properties of the 3-phase separators and the flotation cell. The 3-phase separators could be improved by adding separation plates, and the flotation cell could be optimized by using simulation methods.

3.9 Digital twin of an exemplary water treatment process in the oil & gas industry: Report

A report was created on how a digital twin of water treatment plants in the gas and oil industry was created by using two simulation methods. A digital twin is a simulation of complete plants that can help optimize their performance. The first simulation method was process simulation, which used process simulation software to model the plant behaviour on a large scale. This kind of software can simulate different types of plant components using scripts. The second simulation method was using CFD simulation, to model the detailed areas of interest, such as water/oil interfaces. CFD software can capture the effects of fluid dynamics and phase interactions in the plant. The combination of these two methods improved the simulation model of the water treatment processes. The simulation approach enabled different optimization strategies for the plant to improve the efficiency, quality, and safety of the plant operations.

3.10 Modelling of CQAs based on CPPs: Report

A report was created on the separation of the gas phase and oil residuals from the water. Water is frequently used in secondary recovery methods in oil and gas production facilities. This results in a lot of water that needs to be managed. The main process of separating the water from the oil and gas phases happens in big settling vessels. These big units have various parameters that influence the separation efficiency. Effects on the overall separation performance may be assessed by using sensitivity analysis on python models that simulate such equipment.

Different simulations were carried out to determine the plant parameters and the optimal plant behaviour and control. The results were collected and provided in this report. The parameters were divided in different areas based on their impact on the water quality. The oil residues were measured to calibrate the simulations, but the measuring equipment needed further adaptation. The critical units in the oil-gas separation process were identified and studied in more detail. Python scripts and process simulation were used to show the gravity settling effect on the oil-water separation. CFD was used to simulate the oil-water separation and the oil dispersion. A population balance approach was developed to predict the oil dispersion.

3.11 Alpha version of the HYDROPTICS sensor platform prototype

This refers to the alpha version of the Hydroptics platform, its individual measurement modules, and its assembly.

The platform aims to quantify the oil-in-water content and the total suspended particles in water using novel measurement techniques. It is linked to previous deliverables that discussed the technology of the measurement techniques tested in Hydroptics. Two major parameters shall be quantified with the Hydroptics platform: first, the Oil-in-Water content, and second, the total suspended particles. To do so, a Dual Frequency Comb Spectrometer (DFCS) and a Dual-DFB-QCL based setup will be evaluated for the oil content. The second parameter will be measured using Hyperspectral Imaging (HSI) and a fluorescence-based method.

Due to the complexity of the novel measurement techniques, ancillary devices are necessary.

The ancillary devices include ultrasound transducers for cleaning and guiding the particles, and for the infrared measurements, a sample preparation module for extracting and enriching the oil.

The outcome of the previous tasks was the successful demonstration of the underlying measurement principle and the functionality of the ancillary devices. To build a sensor platform, the modules and the devices were integrated and combined. The platform was installed in 19” wide modules, which are common and standardized in industrial, scientific and telecommunication equipment. The platform was protected against splash water and dirt and was easy to transport.

An overview of the stepwise assembly, the achieved figures of merit and the sensing unit in general was provided. The second iteration of lasers were tested and implemented in the measurement modules during the alpha testing.

3.12 Alpha version of the HYDROPTICS sensor platform prototype: Demonstration

A brief overview of how the alpha version of the Hydroptics sensor platform integrates the research results from previous work into a common platform was created. This includes modules for particle sensing, including a hyperspectral imaging system, fluorescence spectrometer and an ultrasound assisted flow-cell, allowing trapping, and focusing of particles. For oil-in-water analysis, an automated liquid/liquid extraction system suitable for both batch-wise and continuous operation is installed. For quantification, a dual-DFB QCL light source and a Dual Frequency Comb Spectrometer were tested and compared in course of the following alpha testing. The components were tested in-house by the project partners. The demonstrator was assembled and delivered for laboratory evaluation. The alpha testing included stability and integration testing to determine component suitability for the Hydroptics beta platform, which will be subsequently assembled, and field tested in industrial environments.

Figure 13: HYDROPTICS alpha platform.

3.13 Frequency Combs spectrometer testing

The spectrometer for Hydroptics was assembled and tested. A dual-frequency comb spectrometer is a device that uses two lasers with discrete and equally spaced frequencies to measure the spectrum of a sample. The two lasers interfere on a detector and produce a signal that contains the absorption information of the sample. This technique is fast, accurate, and broadband, and can be used for various applications in molecular spectroscopy.

The specific spectrometer used the current technology for the alpha version of the sensor. The demonstrator passed all the tests and the criteria, except for the lasers' spectra, which were slightly wrong. This was decided to be fixable with new lasers. The spectrometer had a laser control unit, a laser module, and an acquisition unit. It could be controlled from a distance or directly. The lasers were produced by ALPES for this project. They emitted light in the mid-IR range and spanned a range of 50 cm^{-1}. The spectrometer was stable as per specifications.

3.14 Report on the results of the Frequency Combs spectrometer testing

This report of the test results of a dual-frequency comb spectrometer for the Hydroptics platform, for process optimisation in the oil industry. The spectrometer uses lasers provided by ALPES, which are based on the specifications as described in the novel light sources section previously.

A description of the assembly and testing of the spectrometer is given, which consists of a laser control unit, a laser module, and an acquisition unit. The spectrometer was assembled using the specifications derived from previous work and was tested to meet those specifications.

Some challenges and solutions were encountered in the testing, such as the slight redshift of the laser frequency, which will be corrected with the following generation lasers. It is concluded that the spectrometer was successfully assembled and tested, and that it can be controlled via an external connection or its own client.

An overview of the test results and the performance of the spectrometer is provided, which emits light in the mid-IR range 1,350 cm^{-1} and spans a range of about 50 cm^{-1}. The instrument stability was within the given specifications as required.

3.15 Beta version of the HYDROPTICS sensor platform prototype Demonstration

A demonstration took place to test the Hydroptics sensor platform, which measures oil and particles in water. A new prototype was designed, as the alpha prototype was unavailable. The new platform consists of three individual prototypes: the so-called “TUW”, “SAL”, and “TUPRAS” modules. The TUW prototype included the liquid handling system and the infrared sensing modules for the oil-in-water analysis. The SAL prototype contained the imaging systems and the ultrasound manipulation for the particle analysis. The TUPRAS prototype consisted of a commercially available Fourier-Transform infrared (FTIR) spectrometer for the spectral characterisation of refinery products. Each prototype was tailored to the specific needs of the testing site. The text also reports how the prototypes were installed in 19” wide modules, which are common and standardized in industrial, scientific and telecommunication equipment. Each prototype was tailored to the specific needs of the testing site, and all were protected against splash water and dirt, while been made easy to transport. The prototypes were designed, built, and assembled successfully. They will be discussed briefly in this demonstrator section below.

3.16 Sensor platform prototype testing

The three TUW, SAL and TUPRAS prototypes were designed, built, and assembled using a modular approach. The following are the key points of the testing of said prototypes.

- Each prototype had a different system to analyse the process water stream for oil and hydrocarbons.
- The prototypes were tested for safety and performance before being shipped to OMV and TUPRAS for field testing.
- The TUW prototype had four modules: Electronics & PC Rack, Aquarius Rack, Dual-DFB Rack, and Liquid Handling Rack.
- TUW prototype uses infrared spectroscopy to quantify oil and correct for water interference.
- The Aquarius Rack and the Dual-DFB Rack used quantum cascade lasers (QCLs) for infrared spectroscopy as seen in the figure bellow.

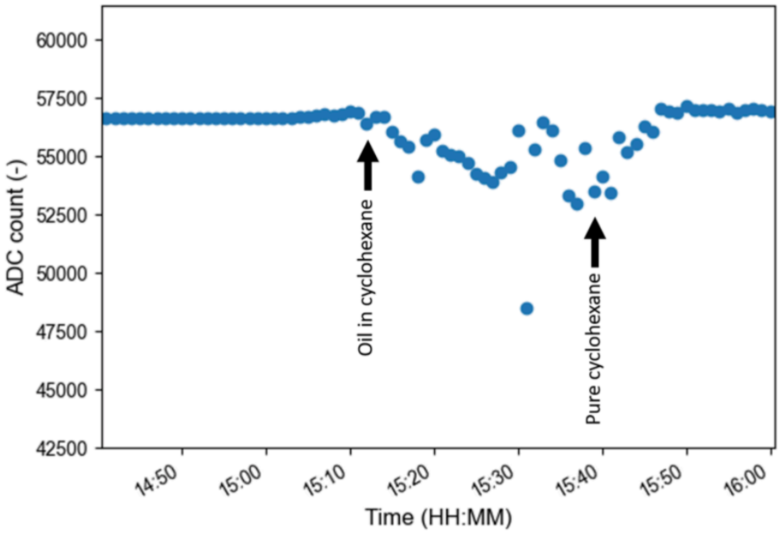

Figure 14: Example of measurement with the Aquarius Rack

- The Dual-DFB Rack had two QCLs emitting at different wavelengths to correct for the interference of water.
- The Dual-DFB setup showed better results than the single DFB setup when water was present in the cyclohexane solvent.
- The QCLs were characterized and calibrated using tetradecane and oil as model analytes.
- Spectral information of the Hydrocarbon mixture can be extracted using the TUPRAS prototype.
- For Cyclohexane, a path length of 0.1 mm is the limit.
- For TCE, a path length of 1.4 mm is still practical.
- Liquid Handling Rack performs extraction and separation of oil and water.

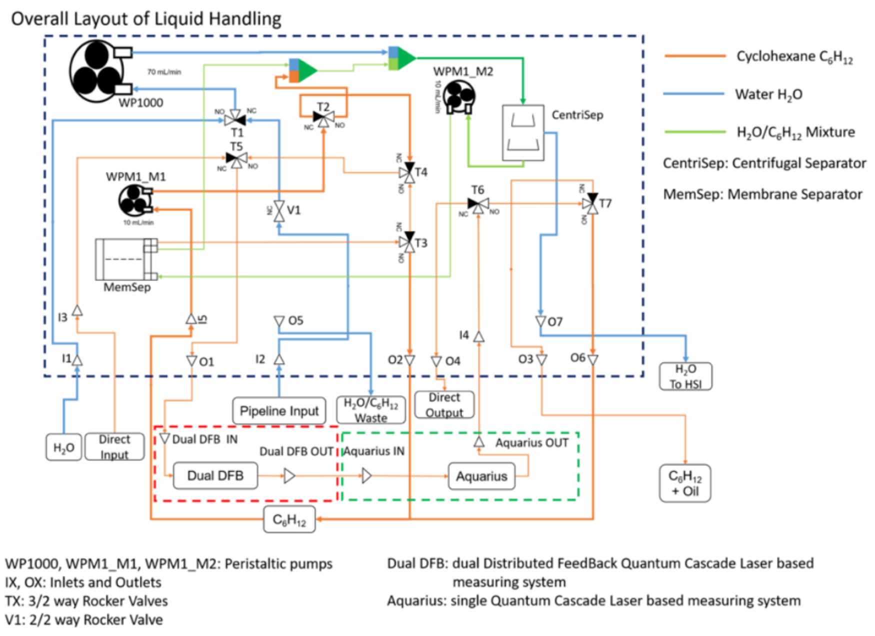

Figure 15: P&ID of the liquid handling system with descriptions of the components

- TUW prototype was tested and shipped to OMV for field testing.
- Particle sensing system uses different imaging techniques and ultrasound manipulation to analyse particles and oil droplets.
- WLI module can distinguish colours of particles, FLI module can detect oil droplets.
- SAL prototype was tested and shipped to OMV for Pilot I test phase.

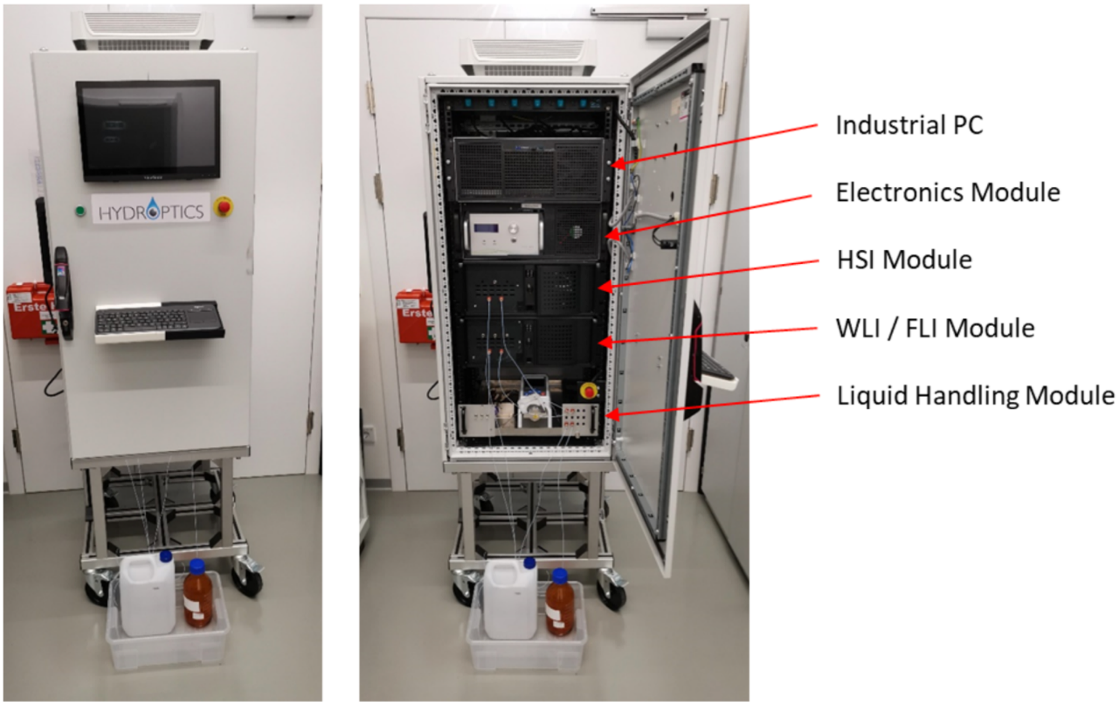

Figure 16: Front view of particle sensing system with closed front door (left) and with open front door.

3.17 Beta version of the HYDROPTICS sensor platform prototype

As mentioned above, the new version of the sensor consisted of the following 3 independent prototypes: the TUW prototype, the SAL prototype, and the TUPRAS prototype

The TUW prototype included a liquid handling system, where the centrifugal separator was located for both the extraction of process water with cyclohexane and their separation. The cyclohexane output was connected to a membrane separator for the removal of residual water followed by 2 IR sensing modules. One contained the dual-DFB setup and the other a single laser module that was already applied in the EU project Aquarius.

The SAL prototype included a liquid handling system to transport an aqueous sample stream to a flow cell for the particle analysis using a white light imaging, fluorescence light imaging or hyperspectral imaging in combination with ultrasound particle manipulation.

For the TUPRAS prototype, it was impractical to use the TUW prototype since the limited tuning range of the lasers offered no information about the chemical properties of the measured oil. TUPRAS was more interested in knowing which fraction is leaking into the cooling water and not how much. Thus, either a tuneable laser was required or a commercial infrared spectrometer. A commercially available Fourier Transform InfraRed (FTIR) spectrometer with a custom flow cell and liquid handling system was adapted and used.

A modular approach was pursued. This enabled tailoring the different prototypes to the needs required to perform the necessary measurements. Additionally, since the modules of SAL were still at their premises, they were able to continue working on their system, independent from the works of TUW to design a new prototype.

The individual module prototypes were connected, to obtain the information provided by each individual prototype. The TUW and SAL prototype were then be tested together and sent to OMV for the Pilot I phase. Both devices also passed a safety inspection requested by OMV.

Due to the limited time and the difficulty of sending such a complex device to Türkiye, only the TUPRAS prototype was sent there to perform the experiments of interest. It offered all functions needed to complete measurements of their samples.

The 3 prototypes were successfully designed, built, and assembled.

A prototype was tested to measure hydrocarbon spectra with different solvents. One solvent had a lower limit of detection but a shorter path length than the other. The other solvent was toxic and banned in some countries, so the first one was preferred.

This also illustrated the need for a tuneable laser emitting in spectral ranges of interest. Due to the higher power of lasers compared to a commercial infrared light source, higher path lengths could be used, thus enabling detection at lower concentration.

3.18 Preparatory activities for pilot demonstration

This preparatory activities for pilot demonstration of the HYDROPTICS project, aimed to develop and test a measurement platform for oil and particles in water in real industry settings. This required the involvement of two industrial partners, OMV and TUPRAS, for the two pilot tests at their sites.

The preparatory work done in the task “Preparatory activities for pilot demonstration” to select and prepare the test sites and sampling points for the pilot tests. The test sites had to provide safe and stable operating conditions and the required infrastructure for the operation of the HYDROPTICS platform. The platform consists of two modules: Module A for oil-in-water measurement, and Module B for particle and droplet measurement.

The challenges and solutions encountered in the preparatory work, such as the low levels of suspended solids in the OMV facility, which required a test skid for additional solids spiking. The report concludes that the test sites at both OMV and TUPRAS were made ready for the field trials of the HYDROPTICS platform.

3.19 Hydroptics Pilots:

The reports bellow provides an overview of the field testing at the sites of the two industrial partners:

- Pilot I at a water treatment plant operated by OMV in Austria
- Pilot II at a refinery operated by TUPRAS in Türkiye

One of the main goals of the HYDROPTICS project was not only to develop a measurement platform, but also to perform the assessment of functionality and figures of merit in real industry settings. The field testing at the sites of the two industrial partners OMV and TUPRAS allowed to do so.

The following reports contain information on the two pilot tests that took place at the sites of OMV and TUPRAS, respectively.

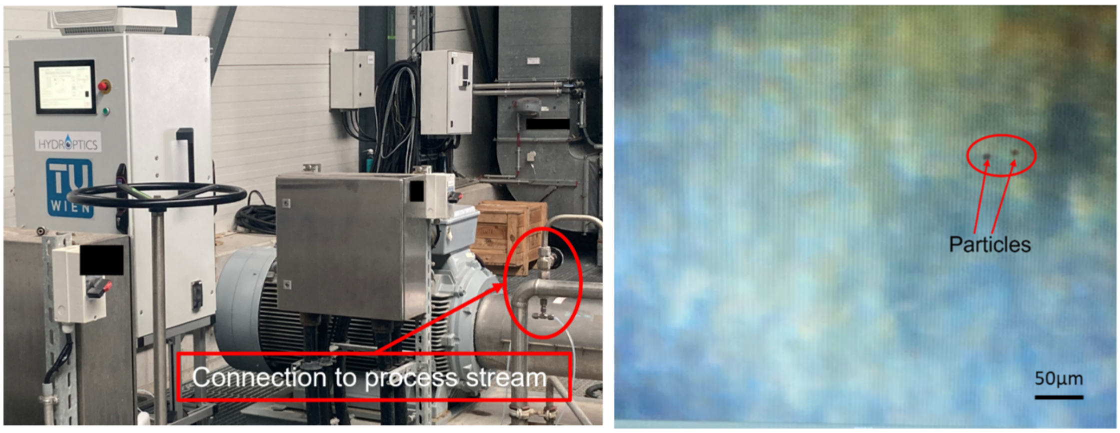

Figure 17: Particles in OMV process stream in the Flow cell of Module B (right), Hydroptics Oil-in-water module connected to the process stream of OMV WTP (left)

The first pilot test was conducted at OMV premises, where the HYDROPTICS platform prototype was tested. The platform consisted of two modules: Module A for oil-in-water measurement, and Module B for particle and droplet measurement. The platform uses various techniques, such as centrifugal separation, membrane separation, ultrasound manipulation, hyper spectral imaging, white light interferometry, and fluorescence imaging, to analyse the water quality and provide data for oil/water separation optimization.

The second pilot test was conducted at TUPRAS premises, where an IR-based at-line analyser was used to give information on oil leaks in downstream refinery operations. The analyser used two spectral regions (1,360-1,410 cm^{-1} and 1,470-1,520 cm^{-1}) to identify the type and source of the leaked products. The analyser was installed in the cooling water system, where water is passed through heat exchangers to chill the hydrocarbon products. The analyser has a limit of detection of ~80 ppm in cyclohexane, which is equivalent to 5 ppm in oil-in-water.

3.20 Successful pilot I completion

The report described the work done within Pilot I - OMV Pilot (Austria) of the HYDROPTICS project, which aimed to develop and test a measurement platform for water quality in the gas and oil industry. The Hydroptics Sensing Platform (“Prototype”) was transported and connected to the process stream at the OMV Water Treatment Plant. The Prototype was piloted for six weeks, providing data acquisition over several hours per day. The project faced a major setback and a time delay due to the termination and liquidation of QRT, the project partner responsible for the assembly of the sensor systems. The prototype had to be reconstructed by TUW and SAL and split into two modules: Module A for liquid handling and oil-in-water analysis, and Module B for particle measurement. OMV had to deal with all HSSE concerns regarding the prototype and obtained the authorization for testing it in CW 42.

The report also described the results and the lessons learned from the field test at OMV premises. The field test successfully demonstrated the functionality of the prototype and the measurement principles of the sensing device in an industrial environment. The field test also showed the importance of testing the prototype under field conditions for both the technology provider and the interested party, as laboratory testing could not substitute for onsite testing. The field test involved environmental and process conditions that could not be recreated in the laboratory, as well as HSSE topics that were not applicable for laboratory surroundings.

Figure 18: Modules A & B connected and measuring OMV WTP process stream (left); Particles, air bubbles and oil droplets on the optical window (right).

The results of a field test of two prototype modules for measuring oil and particles in water at the WTP Schönkirchen, a facility with multiple sampling points. The modules are designed to use different techniques to analyse the water quality and provide useful data for oil/water separation optimization.

Module A used a centrifugal separator and a membrane separator to measure oil-in-water concentration. It faced a problem of reduced operation time due to a change in the process stream composition, which was solved by adjusting the flow and revolution rate. It showed sensitivity to different oil-in-water concentrations but required data postprocessing and calibration to align with reference measurements.

Module B used a combination of ultrasound manipulation, hyper spectral imaging, white light interferometry, and fluorescence imaging to measure particles and droplets in water. It did not operate the hyper spectral imaging system due to the long integration time and the heating effect on the flow cell. It confirmed the low amount and size of particles in the process stream but failed to image them in the volume of the flow cell due to the heterogeneity and the low optical contrast.

Module B could detect particles, droplets, and bubbles on the surface of the optical window, and provided size distribution data on the oil droplets, which was useful for oil/water separation optimization. However, the ultrasound manipulation showed some guiding effect for the oil droplets but was not able to keep them in the focus of the imaging system. This was due to the size distribution of the oil droplets and the presence of different particles and droplets with different acoustic contrasts and size distributions in the process water stream.

In summary, the report presents the results and challenges of each pilot test in different scenarios. For the first pilot test, Module A faced a problem of reduced operation time due to a change in the process stream composition, which was solved by adjusting the flow and revolution rate. Module A showed sensitivity to different oil-in-water concentrations but required data postprocessing and calibration to align with reference measurements. Module B did not operate the hyper spectral imaging system due to the long integration time and the heating effect on the flow cell. Module B confirmed the low amount and size of particles in the process stream but failed to image them in the volume of the flow cell due to the heterogeneity and the low optical contrast. Module B could detect particles, droplets, and bubbles on the surface of the optical window, and provided size distribution data on the oil droplets.

3.21 Successful pilot II completion

This report described the main goal of the HYDROPTICS project, which was to develop and test a measurement platform for water quality in the gas and oil industry. The project had two phases of testing at the sites of two industrial partners, OMV and TUPRAS. The first phase tested the prototype of the platform at OMV premises using TUPRAS water samples, representative of cooling water used in downstream refinery operations. The second phase tested the ability of an IR-based at-line analyser to detect and identify oil leaks in the water at TUPRAS premises. The analyser used two regions of light, 1,360-1,410 cm^{-1} and 1,470-1,520 cm^{-1}, to measure the type and amount of oil in the water. The analyser could be used with the platform to quickly respond to leaks and improve the efficiency of the water treatment.

The report also describes the work performed, the results obtained, and the potential improvements for the analyser and the platform. The work involved analysing the samples from different refinery plants and streams in the IR-based at-line analyser and determining the effective regions for hydrocarbon samples. The results showed that the analyser had a limit of detection of 80 ppm in cyclohexane, which was equivalent to 5 ppm in oil-in-water, considering an extraction factor of 18. The improvements included adapting the analyser to sudden changes in the oil content and recalibrating the analyser when the oil content deviated from the nominal value. The report was part of the deliverable Pilot II – TUPRAS Pilot (Türkiye), which also included the field testing of TUPRAS water samples with the HYDROPTICS platform at OMV premises for oil-in-water quantity analysis, as part of pilot I.

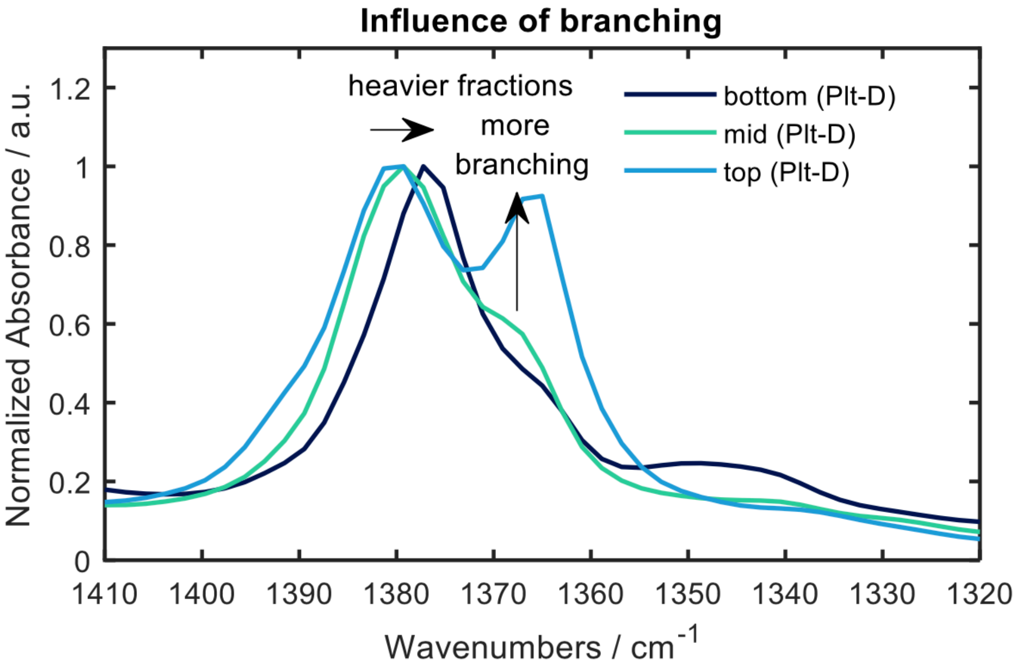

Figure 19: Influence of branching on the symmetric CH3 deformation vibration. Samples were taken at different locations of a rectification column

The report presents the results and challenges of each pilot test in different scenarios.

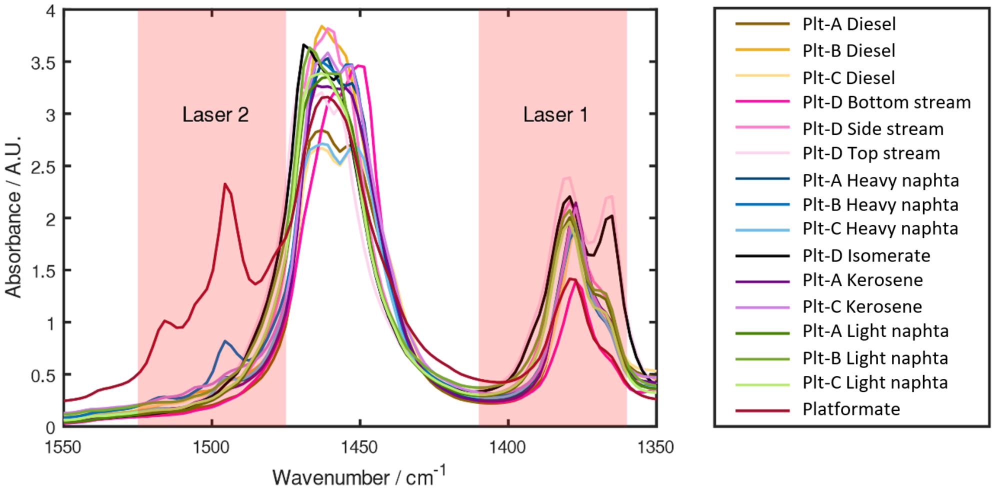

Figure 20: Spectra of the samples acquired in the second pilot with the spectral regions of interest (as specified in D3.2) marked in red.

For the second pilot test, the analyser was used to estimate and point out the candidate leak points, by studying the characteristics of the leaked oil samples. The analyser performed a calibration series of different concentrations of an aromatic-rich product and calculated the limit of detection for each peak in the spectra. The analyser also investigated the spectral differences exhibited by products at different points in the process, such as the rectification of products after isomerization, the presence of aromatic compounds, and the distinction of products from different plants and fractions. The analyser also used tuneable laser sources and machine learning to enhance the identification of different products.

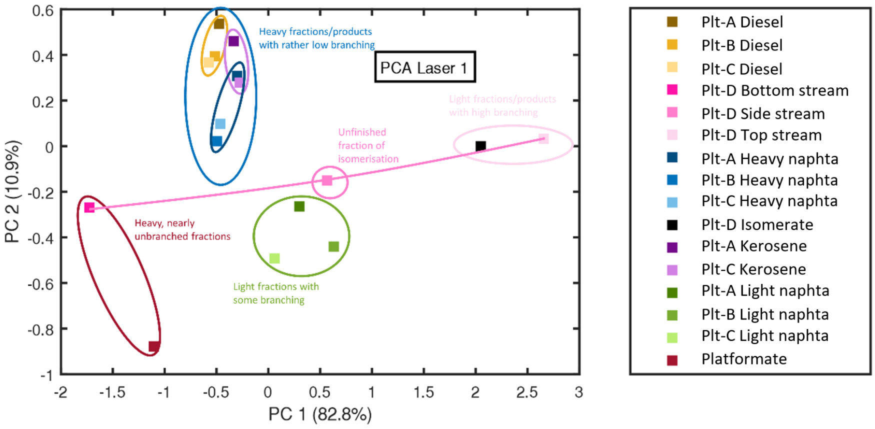

Figure 21: PCA of the spectral features in the first region of interest (branching and chain length).

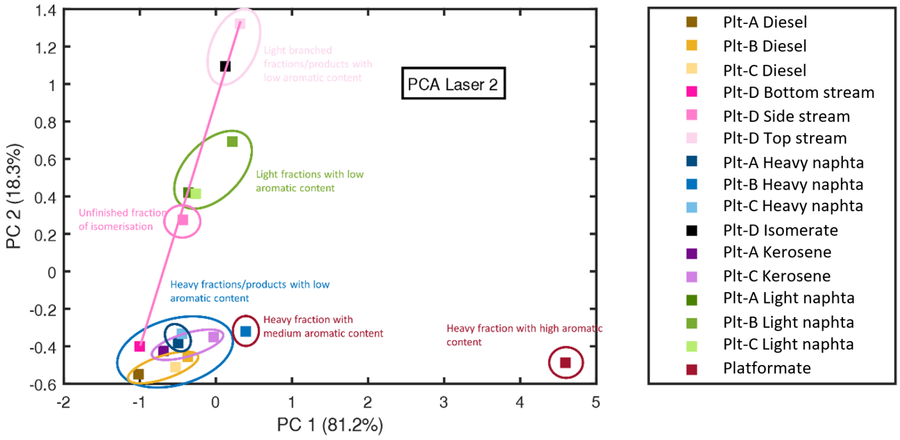

Figure 22: PCA of the second spectral region of interest (aromatic content and chain length).

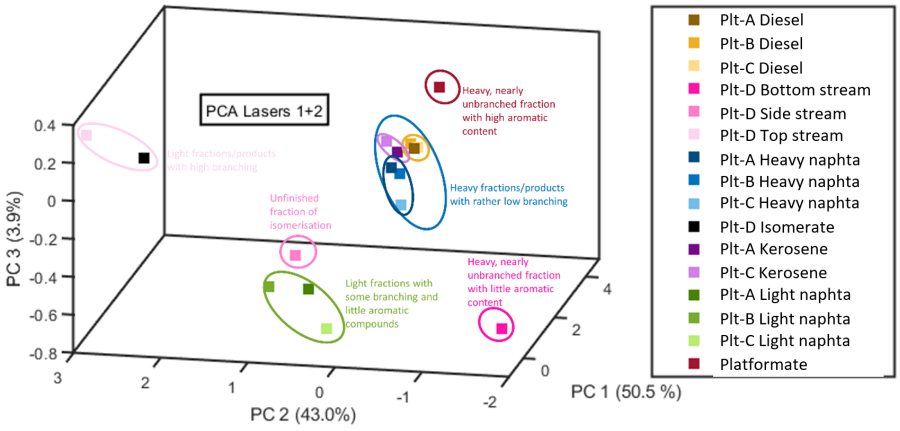

Figure 23: PCA of the full spectral information in both regions of interest (branching, aromatic content, and chain length).

In summary, the report demonstrates the capability of the HYDROPTICS platform and the IR-based at-line analyser to identify and deliver information on oil and particles in water in real industry settings. The second pilot test was conducted at TUPRAS premises, where an IR-based at-line analyser was used to give information on oil leaks in downstream refinery operations. The analyser used two spectral regions (1,360-1,410 cm^{-1} and 1,470-1,520 cm^{-1}) to identify the type and source of the leaked products. The analyser was installed in the cooling water system, where water is passed through heat exchangers to chill the hydrocarbon products. The analyser has a limit of detection of ~80 ppm in cyclohexane, which is equivalent to 5 ppm in oil-in-water. The report also discusses the potential improvement steps and applications of the devices for oil/water separation optimization and quality control.

3.22 Novel monitoring device performance

The prototypes were implemented and evaluated in the fields. The first prototype successfully monitored and separated the oil-in-water concentration and the particles in the process stream. The second prototype was able to distinguish and measure the water, oil and air bubbles using the fluorescence module. Both prototypes could be connected in a single system to obtain both information of the oil-in-water content as well as some information of the particles. However, some improvements are still necessary to make this system stable enough to be used as an online analyser. This clearly shows the importance of field testing as the conditions of the field could never be simulated in the laboratory. The third and final prototype was able to analyse the hydrocarbons in the samples but would require a laser-based analyser to detect lower concentrations of oil-in-water and could be used in the future to quickly react to leaks in the cooling water system, reduce down-time and improve efficiency.

3.23 Optimised process algorithms

This report describes how optimization strategies from WP6 Task 6.4 were used to validate test facilities. It explains how the layout of these facilities was examined to find the best position for validating the algorithms. Additionally, it identifies the water outlet of the 3-phase separator as the optimal position for validation since it exhibits the most significant dynamic changes during operation. Finally, it provides a forward-looking perspective on future steps in the validation process. It also reports the development of a predictive model for test facility events using sensitivity analysis. This shows how optimization potentials are identified and validated by parameter changes and measurements. while also giving a framework for measuring regimes and future optimization efforts.

4 Conclusion

The HYDROPTICS is a project that has successfully achieved its goals and targets, having a robust public presence and therefore significant impact on the relevant areas of interest.

This impact can be better illustrated by comparing its achievements with the Expected Impact Points defined on the call that Hydroptics was selected to be funded for:

Increased competitiveness of the European production industry and significant contribution to the digitization of European industry (EI-1): Hydroptics contributes to increased competitiveness of the European production industry through increased digitization in several ways. The productivity of oil production in almost depleted oil fields relies on the reinjection technique which requires large quantities of water being recirculated for oil production (extraction). For optimization of this process accurate information on residual oil and particle content in the circulating process water is needed. Equally important is detailed knowledge on mass and energy transport in the industrial water purification plant. Hydroptics addresses these challenges through the development of a novel on-line analyser based on advanced photonic devices for quantitative and qualitative analysis of oil and particles in the process water and by gaining insight in the operational aspects of both the analyser as well as the industrial plant. In this regard digitization is required throughout. Machine learning algorithms are be used and digital twins were developed for detailed simulation of core operational units of the analyser as well as the plant itself. These developments increase the competitiveness of the EU production industry significantly and drive the oil industry towards more digitized operation.

Contribution to innovation capacity and integration of new knowledge (EI-2): Each of the HYDROPTICS prototypes compartments have a strong innovation touch to them increasing their spectroscopic and analysis capabilities. In particular, the laser modules will have first time demonstration of integrated chip-scale dual laser systems that will decrease the footprint of the entire system and ease the utilization of modern frequency comb spectroscopy also for monitoring of industrial processes. The hyperspectral imaging module will have the capability to precisely measure particles in industrial water-oil emulsion. Making such information available in real-time and its combination with process data and insights obtained from digital twins will set the ground to create new knowledge. For sample preparation, a novel type of centrifugal separator will be used to better perform the phase separation of the liquids after liquid -liquid extraction, and continuous detection of oil-in-water will be implemented. All these modules are integrated in a single rack with a common interface to readout all the units with one window. Hence, the impact on innovation and integration of new knowledge is very present and significant.

New market opportunities, competitiveness and growth of companies (EI-3): With the developed technologies, HYDROPTICS Consortium is already monitoring also other markets than the oil industry. In particular, one can envisage using dual-comb spectrometers for hazardous gas detection in fabrication facilities, milk analysis, protein dynamics analysis. Moreover, with the hyperspectral imaging module, one can go much further and detect not particles and oil droplets, but also their chemical composition, and with machine learning algorithms make fast and rugged qualitative analysis allowing for clustering and segmentation. The developed integrated chip-scale beam combined dual QCL source has a huge potential to become a generic product, that will be used in all the spectroscopic applications requiring two or more wavenumber lasers (gas detection with background, etc.). Hence, the impact is still very relevant.

Quality of life, health and safety of the citizens (EI-4): Automated quality control of process water will set the ground for running oil production more cost effective and to reduce adverse impact on the environment. HYDROPTICS prototype will allow for better water purification and filtering of the process waters, thus reducing the current need for make up chemicals to be added to the water. As process waters from up- and downstream processing (oil production and refining) are finally being released to the environment, better control on the overall process has an immense impact on the environmental safety. The HYDROPTICS spectrometers can be used in general for water purification facilities. We believe that the prototype will open new opportunities with applications also beyond water purification in the oil industry.