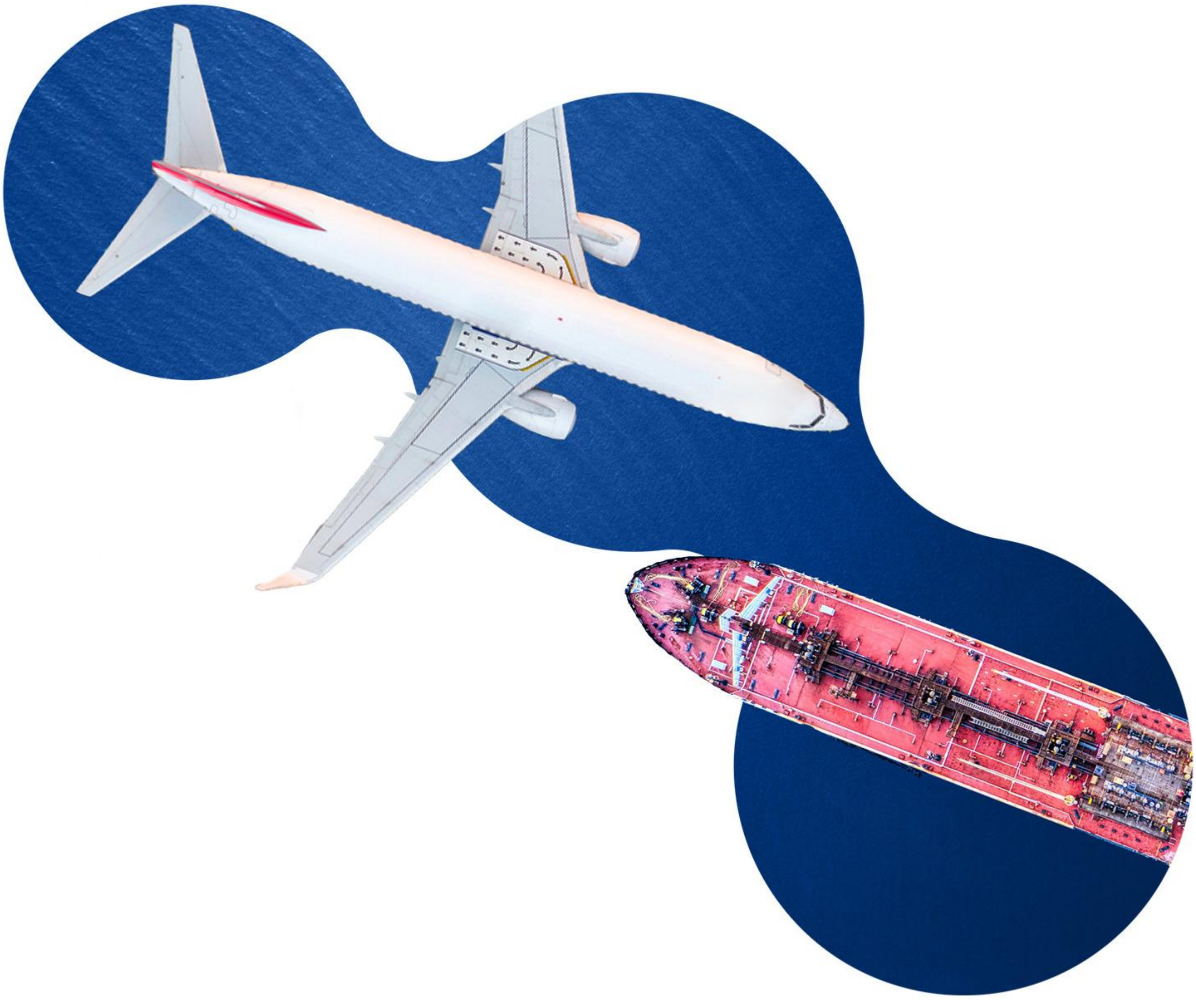
COCPIT

D3.2

First draft of reactor based on kinetics and feed characteristics from literature and campaigns led on different continuous HTL systems



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Statement of Originality

This deliverable contains original unpublished work except where clearly indicated otherwise. Acknowledgement of previously published material and of the work of others has been made through appropriate citation, quotation or both. This project has used a standard methodology already developed in BECOOL project (Grant Agreement number: 744821), following EU recommendations. Ad hoc modifications were added to comply with the Grant Agreement conditions for COCPIT (Grant Agreement number: 101122101).

Disclaimer of warranties

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Executive Summary

The current deliverable (D3.2), is a demonstration model for the first draft of the continuous reactor tailored for the hydrothermal liquefaction of microalgae. In this report, the current status of the built unit will be presented and described. The purchased components will be listed, the manufactured parts will be presented, along with the assembly and operation of the overall unit.

The study and design phases of the continuous unit is already presented in a previous deliverable; D1.2: Technology specifications, requirements. For any info regarding the selection, sizing, design and manufacturing of different equipment, kindly refer to D1.2, part 4: Continuous HTL Reactor for SAF Production.

This report is divided into two main parts:

- Components and unit assembly
- Operating protocol

1. Components and Unit Assembly

In this section, the different components purchased, designed and manufactured, which are previously detailed in D1.2 will be presented, including their characteristics and specifications. To recall the overall unit's, the P&I diagram is shown in Figure 1 below:

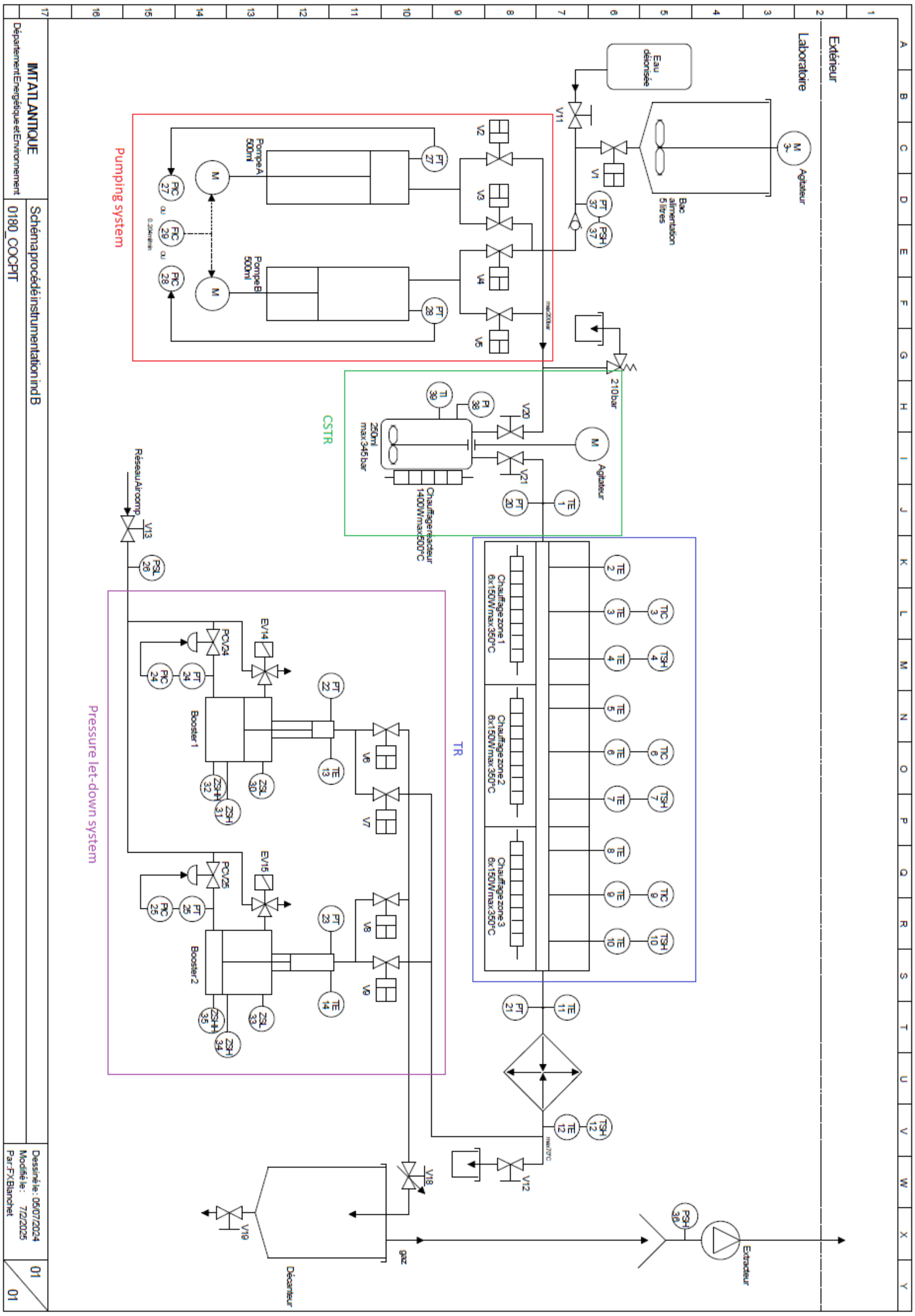

Figure 1: P&I diagram of the continuous unit

In brief description of the process, microalgae slurry (20 wt.%) is prepared and stored in a continuously mixed 5 L feed tank. Another tank contains deionized water for starting up the system to reach its steady state. The high-pressure double-piston pump injects the microalgal slurry continuously, with a pre-set flow rate, to the CSTR. The ambient temperature feed is thermally shocked in the CSTR, resides for the required time which may vary between a couple of minutes up to 30 mins, depending on the CSTR configuration and the flow rate, and then continues its flow to the tubular reactor, where the rest of the residence time is spent there, ranging between an additional one minute, up to 30 mins, depending on the flow rate and the configuration of the TR. After exiting the TR, the products are cooled in the cooling system to reach a temperature below 70 ℃, after which the cooled products enter the pressure let down system (double air-hydraulic boosters). These boosters regulate the pressure of the system, their alternative operation ensures continuous depressurization of the products, which are vented into the collecting tank, separating the liquid products from the gaseous phase.

1.1.Feed Tanks

Two different feed tanks are manufactured at IMT's work shop and installed in the continuous unit. The microalgal slurry's feed tank has a volume of 5 L, which is continuously mixed to avoid any biofilm accumulation on the tank's walls, and ensure the homogeneity of the slurry throughout the processing time which may last over tens or hundreds of hours. The other tank is a de-ionized water tank, in which the system is started using de-ionized water for filling the components, pressurizing, heating and reaching the steady state process conditions (pressure, temperature and flow rate). Figure 2 shows a photo of the two feed tanks, mounted on the frame of the continuous unit. The left tank with a motor stirrer is the microalgal slurry feed tank, the smaller one on the right is the de-ionized water feed tank. Both tanks are connected to a solenoid valve, shown in Figure 3, allowing allows the user to choose what tank shall feed the system.

Figure 2: Feed tanks

Figure 3: Solenoid valve for tank selection

1.2.High Pressure Pump

The high-pressure pump is required for precisely controlling the flow rate of the feed at while maintaining a pressurized medium. A double piston ISCO pump; SYRIXUS 500XV was purchased. The characteristics of the pump are shown in Table 1.

Table 1: SYRIXUS 500XV specifications.

Capacity (mL) (per pump)	507
Flow range (L/h)	0.00006 – 12.24
Flow accuracy	0.5% of setpoint
Pressure range (bar)	0.7 - 345
Standard pressure accuracy	0.5%
Standard plumbing ports	3/8” NPT
Dimensions (cm)	102 × 27 × 47
Continuous flow range (L/h)	0.00006 – 7.92
Maximum viscosity (cP)	500

The pumping system consists of two piston pumps, alternatively operating (filling and pumping) to ensure flawless continuous operation for extremely longs runs. The alternative operation of the double pistons (pump A and pump B on Figure 1) is controlled by a set of pneumatic valves. The whole assembly is connected and controlled by a main panel that provides quite flexible control over all the parameters, including but not limited to: closing and opening the pneumatic valves, setting up a pumping protocol with different flow rates and different pressure levels, utilizing single piston or alternating the two pumps, setting up minimum and maximum limits for the piston travel distance, etc.

Figure 4 shows the control panel of the pumping system. Figure 5 shows recto-verso photos of the pumping system mounted and assembled on the continuous unit’s frame. The double piston pumps, the pneumatic control valves, and the control panel are visible in the photo.

Figure 4: Main control panel of the pumping system

Figure 5: Recto-verso photos of the pumping system mounted on the frame of the continuous unit

1.3.Continuous Stirred Tank Reactor (CSTR)

The design and selection steps of the CSTR for the continuous unit are previously explained in deliverable D1.2. The CSTR was purchased from PARR Instruments. The unit features a heating oven, able to reach temperatures up to 500 ℃. In the oven, the reactor, which is made of 316 stainless steel is placed. The reactor is able handle the maximum temperature of the oven, at elevated pressures up to 345 bars. The internal diameter of the reactor is 2.5’’ (63.5 mm). Three different vessels were purchased, varying the internal height to feature three different reactors with 250-, 150- and 100-mL volumes (Figure 6). This combination provides maximum flexibility for the usage of the CSTR under different heating rates and different flow rates. The feed in the reactor is continuously stirred through a magnetic coupling stirrer, with a variable rotational speed. The CSTR unit is shown in Figure 7. The whole system is controlled through a main panel, allowing full control of all the different parameters (Figure 8).

Figure 6: 100-, 150- and 250-mL reactor vessels; same outer geometry, however, the internal diameter differs to obtain the desired volume

Figure 7: CSTR Unit

Figure 8: CSTR Reactor controller

1.4. Tubular Reactor (TR)

The manufacturing process of the TR was conducted in the workshop of IMT Atlantique based on the proposed design detailed and explained in the previous deliverable D1.2. The reactor consists of a 0.9 m long tube, with a 14 mm outer diameter, and a 10 mm inside diameter. An aluminium casing is manufactured to accommodate the tube, which is divided into 3 different sections, 30 cm each. In each casing section, 6 heating elements, 150 W each, are embedded, yielding a total of 2700 W heating power in the TR. Each casing heating mode is independent, allowing the full control over a section of the reactor, or the whole reactor at once. Type K thermocouples are planted along the length of the reactor, their rule is crucial in visualizing the temperature gradient along the reactor and validating the temperature stabilization (steady-state mode). Pressure is measured upstream and downstream the TR to check the pressure drop across the system, and to help in detecting clogging issues when occurred. The system is insulated using glass wool for minimizing the heat loss and improving the overall efficiency of the heating process. Figure 9 shows the TR with the connected measurement gauges mounted on the frame of the continuous unit.

Figure 9: Tubular reactor with the inserted thermocouples and pressure transducers

1.5. Cooling System

The cooling system plays a great role in seizing the reaction almost immediately after the products leave the TR spending the desired residence time. This is crucial in avoiding side reactions that decrease the overall yield of the biocrude. In addition, the pressure letdown system (explained in next section) cannot handle fluids at temperatures higher than 70 ℃, thus, the products slurry is shall be cooled before entering it.

The cooling system design is explained in D1.2. The system utilizes a 0.9 meters long pipe, with finned modules installed on it to enhance the heat transfer, is placed just after the tubular reactor. This system is coupled with air-blowing fans to enhance heat dissipation.

Figure 10 shows the manufacturing process of the fins casing done at IMT's workshop.

Figure 10: Fins casing manufacturing for the cooling system

Figure 11 shows the front and back views of the final cooling system after assembly, mounted to the frame of the continuous unit. The front view clearly reveals the fins used to enhance the heat transfer and facilitate heat dissipation, while the back view shows the air-blowing fans inducing forced convection with ambient air.

Figure 11: Front and back view of the cooling system mounted on the continuous unit's frame

1.6. Pressure Let-down system

As discussed in previous meetings (work package leaders, consortium bi-annual meetings), and as explained in D1.2, the initial design proposed for the pressure let-down system consisting of a filter and a back pressure valve (BPV) is changed. During long autonomous operation, this concept is not applicable, due to the solids accumulating in the filter, and clogging it in relatively short times. For this reason, another approach is studied and adopted, inspired thankfully by our partners at Aalborg University.

The new pressure let-down system consists mainly of two air hydraulic boosters. An air-hydraulic booster is a device that uses compressed air to generate high-pressure hydraulic fluid. Essentially, it's a way to get the force of hydraulics without needing a full-fledged hydraulic pump system. In our application, the air hydraulic boosters will be inversely used, they will serve the role of creating the pressure in the system (in the upstream section) through blocking the flow rate provided by the pump, the second role is to reduce the products pressure (in the downstream section) to atmospheric pressure allowing for products collection. In other words, the two air hydraulic boosters will operate alternatively in inverse manner to the double piston high pressure pump.

Two AHB66 air hydraulic boosters were purchased from ENERPAC, having the following characteristics:

Table 2: AHB66 hydraulic air booster specifications

Pressure ratio	1:64
Stroke (mm)	145
Oil volume per stroke (cm^3)	73.3
Air operating pressure (bar)	1 – 5
Oil pressure at 5 bar air (bar)	330
Air consumption per cycle (cm^3 at 6 bar air)	64.1
Air piston diameter (mm)	203
Hydraulic piston diameter (mm)	25
Weight (kg)	16

Figure 12: AHB66 hydraulic air booster

Figure 13 shows the front and back views of the pressure let-down system; air hydraulic booster, pneumatic control valves and limit switches, mounted on the frame of the continuous unit.

Figure 13: Pressure let-down system; air hydraulic boosters, pneumatic control valves and limit switches (front and back views)

2. Operating Protocol

Each of previously detailed component can be operated individually, using a separate control panel of each sub-system. However, for the full flexibility of the control of the continuous unit, and for easing the autonomous operation, a parallel control system is developed. LabView software is installed on a PC dedicated for the continuous unit, and all the subsystems, including feeding, pumping, CSTR, TR, cooling and pressure let-down systems are controlled in one interface. Acquisition systems record store all the data from the system. Graphical interfaces of the components, main control valves, and controlling parameters are shown as live data on LabView.

Figure 14 shows the main screen of the graphical interface of LabView utilized for supervising and controlling the continuous unit.

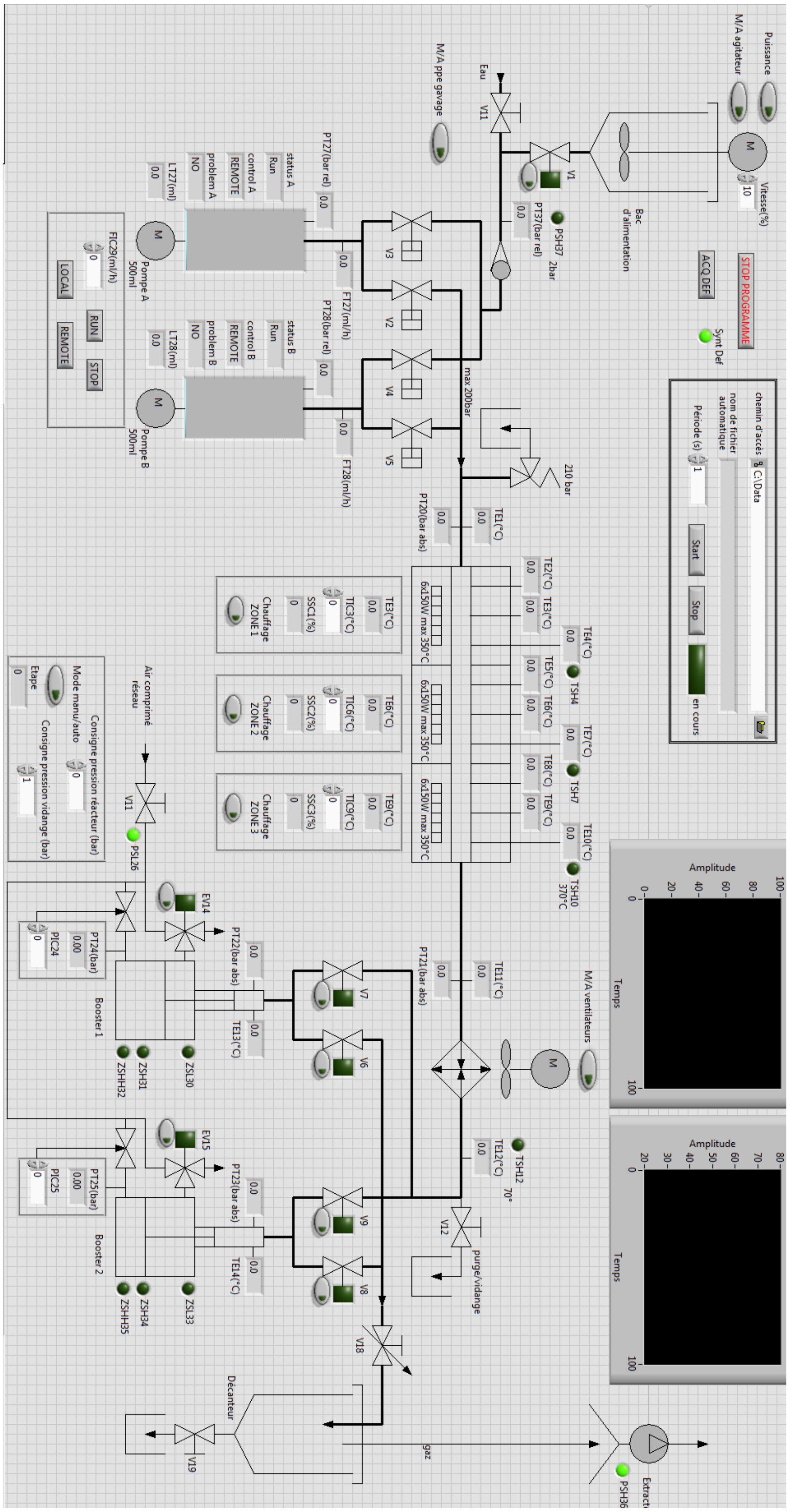

Figure 14:LabView's interface for remotely controlling the continuous unit

The pump can be operated through two different options; local, which requires using the main control panel of the pump, or remote, allowing LabView to interact directly with the pump's operation, specifying the desired flow rate for the process. In remote operation, the pump only regulates the flow rate; the system's pressure is regulated by the pressure let-down system (hydraulic air boosters).
The CSTR temperature can be defined using LabView. A single temperature can be set, or a heating protocol can be programmed. Regarding the tubular reactor, each module (30 cm) containing 6 heating elements can be heated independently.
A button can be triggered for turning on/off the fans of the cooling system. A future approach may be either controlling each of the five fans independently, or applying analogue control (0 – 100% rotational speed) instead of on/off operation.
The pneumatic control of the pressure let-down system is autonomous once the system's pressure has been defined. However, the interface allows access for manual control over each valve. This is essential for emptying the system manually in case any fault (or clog) appears.

Working with elevated temperatures up to 350 ℃ under 200 bars pressurized medium induces high risks. For this reason, several safety features have been installed for ensuring the safety of the users and the equipment:

- The pump has a built-in cutoff relay which immediately seizes the pumping process and vents the excess pressure (if needed) preventing over-pressurizing the whole system.
- A pressure relief valve (210 bars) is installed between the pump and the CSTR.
- Each heating module is protected by a safety relay, cutting the local heating power in case the temperature exceeded 350 ℃ (or the pre-defined value).
- A thermocouple is placed after the cooling system. In case its temperature exceeds 70 ℃ (the maximum allowable temperature to enter the hydraulic air boosters), the heating power is shut automatically, and if needed, a purging valve is connected before the boosters, which may be used to empty the hot fluids and protect the system.
- A safety stop switch is installed on the main power board of the continuous system, which can always be used to cut the power off the system in case of emergencies.

Figure 15 shows front and back views of the continuous unit.

Figure 15: Front and back photos of the assembled continuous unit