Advanced Carbon Capture

for steel industries integrated in CCUS Clusters

Innovation Action

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D2.4 Factory acceptance test results of the CASOH pilot

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Contributors: Marcos Cano (AMIII)

Dissemination level: PU

1 Version log

Version	Date	Released by	Nature of Change
0.1	19/10/2023	Marcos Cano	Initial draft
0.2	20/10/2023	Paul Cobden	Initial draft review
1.0	30/10/2023	Marcos Cano	Final version

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2 Contents

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3 Definition and acronyms

Acronyms	Definitions
AMA	Arcelor Mittal site in Asturias (ES)
BFG	Blast Furnace Gas
CASOH	Calcium Assisted Steel-mill Off-gas Hydrogen
GASLAB	Arcelor Mittal Gas Laboratory
NG	Natural Gas
OGS	General Safety Order
SCADA	Supervisory Control and Data Acquisition
TGA	Thermogravimetric Analysis
WGS	Water Gas Shift

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4 Introduction

The CASOH process under development in the C[4]U project, is based on a high-temperature solid looping system. This process uses packed-bed technology combined with pressure and temperature swing stages. In the CASOH reaction stage, the Cu-based material catalyzes the water-gas-shift (WGS) reaction of the CO contained in the BFG with steam, whereas the CaO-based material reacts with the CO_2 present in the inlet BFG and the CO_2 generated in the WGS reaction. As a result, a decarbonized fuel gas concentrated in H_2 and N_2 is obtained in this stage.

The remaining two reaction stages ("oxidation" and "regeneration") are designed to produce, in concentrated form, the CO_2 stored as $CaCO_3$ during the CASOH stage. During the OXIDATION, the Cu-based material is oxidized with air, this could be done preferably at high pressures to avoid the decomposition of the $CaCO_3$, but can be performed, as in the experiments, at low pressures taking into account the possible decomposition of the material. This reaction stage is very fast and produces a hot stream of N_2 suitable for heat recovery and utilization (for power generation or other uses). The final REGENERATION stage, exploits the unique property of CuO to be exothermically reduced to Cu with a fuel gas, obtaining only CO_2 and water vapour as products. The energy released during the CuO reduction is used to drive the decomposition of $CaCO_3$ into CaO and more CO_2 which allows the start of a new CASOH stage. The full process scheme may contain several additional heat removal stages to close the full reaction loop and other additional steps.

Following the DoA, the present D2.4 document describes all the necessary pre-test work to ensure that the pilot plant can operate as planned, following standard testing and approval regime of prototype to comply with existing permits for operation of pilots in the GasLab site of AMA. This implies the acceptance by AMA and CSIC (beneficiaries in charge of the commissioning of all pilot components) of all pilot elements and all the works carried out by service providers participating in the construction of the pilot plant. The finalization of all the commissioning works presented in this deliverable, marks the beginning of the experimental works, towards long-term tests at TRL7, to be reported in D2.5 and D2.7.

Figure 1: Finalized CASOH pilot plant

This deliverable starts with the description (section 5.1) of the site and the initial civil works to allow the pilot plant construction and the supply fluid lines to the pilot (Blast Furnace Gas, Steam, Natural Gas (NG), Nitrogen, "industrial water", from the factory gas lines of the AMA plant) as well as the new electric works to accommodate the power demands from the pilot. In section 5.2, the process of construction is described by going

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to through the structural elements, reactor and auxiliary elements and the piping, actuators and instrumentation.

A review of the safety analysis (including HAZOP and HAZID) is provided in section 6. The report concludes (section 7) with a description of the commissioning test carried out to allow the imminent start of the experimental works on the pilot, after loading a first batch of active materials, cleaning pipes and ensuring utilities supplies, individual actuators tests, hydraulic check to avoid gas leakages and operation of the gas preheater and off-gas cooling and flaring.

This document is complemented by a compilation (available to project partners at: https://saco.csic.es/index.php/s/YrnExmSW54WPcNd) of all detailed technical specifications and certifications from suppliers of individual pilot components, as well as construction drawings and other detailed services needed to commission the pilot. For operational protocols of the pilot in their different operational modes, we refer to deliverables D2.2 and Addendum to D2.2.

5 ArcelorMittal R&D facilities -pilot plant construction

The following section compiles the information on the pilot plant construction process. Including not only the information of the actual pilot, but also the information of all the associated facilities and utilities required to operate the pilot plant.

It must be noted that, from the work-safety point of view, all the pilot plant construction works by different contractors and service providers followed the established rules applicable inside the industrial site of the Arcelor Mittal plant. The different safety considerations are described in the following sections.

5.1 Laboratory preparation works for CASOH pilot plant.

5.1.1 Selection of the construction site

The selection of the construction site was agreed considering the different impacts associated with the operation of this pilot. ArcelorMittal laboratory (GasLab) already contains several pilot plants that need to operate without mutual interference. Additionally, and due to the impact of other decarbonization projects, the availability of area to construct such a large-scale pilot plant was limited.

Gas treating technologies using fuel gases face challenges when dealing with risks. CASOH pilot plant must deal with three major gases that are either used or generated during the operation:

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- Blast Furnace Gas (BFG): a mixture of N_2, CO_2, CO and H_2 gases generated at the top of the blast furnace and that is used as target to decarbonize in the CASOH process. This gas presents a risk of intoxication and explosion that has to be considered in every stage of the construction.
- CO_2 product: This is a variable gas stream of N_2 and concentrated CO_2 that is generated as product in the CASOH process. These two gases are not inherently dangerous, but high concentrations of them in a closed area can displace the O_2 from the area, reaching potentially dangerous levels.
- H_2 product: This is the N_2/H_2 product gas from CASOH, that at certain concentrations can generate potential explosive areas.

These points were considered for selecting the final location of the pilot plant. Others are for example, the noise impact or the absence of any other industrial pipeline nearby the pilot plant.

Apart from the previous points, as a pilot plant, it is necessary that is as accessible as possible to carry out any future modifications, or to solve as easy as possible the operational problems that could occur during the tests.

The pilot plant was located outside of Gaslab facilities, next to the control room but outside the buildings partially occupied by other Gaslab pilots. This location enabled to create an industrial gas grid for supplying all the necessary utilities for the pilot plant. Being located outside, the pilot plant gas risks were minimized.

Figure 2: Construction site of the pilot plant

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5.1.2 Civil works

After the final definition of the pilot site, it was necessary to prepare the area. Initially all the sections of the pilot plant were marked on the ground to stablish the limits of the pilot plant.

Due to the anticipated weight of the reactor and its main components, and considering the characteristics of the surrounding terrain, the ground was reinforced with concrete foundations. Special reinforcements were done for the reactor location to ensure no terrain movements and proper leveling. Reactor area is the most sensitive area and these reinforcements will help to reduce the risks of terrain movement.

Civil works had to be scheduled considering the different terrain loads that the pilot plant could apply to the terrain and surroundings. To do this, the following terrain configuration was selected:

Table 1: Materials and quantities used for civil works

Parameter	Units
Excavation	45 m³
Aggregate filling material	33 m³
Floor and foundations	Concrete HA-25 (21 m³) + corrugated steel B500S (1,8 t)

To provide sufficient strength to the concrete. The material was left curing 28 days before placing any load on it. All calculations for foundations, were calculated considering the Spanish technical procedure (CTE) including the different supporting loads (reactor, preheaters, flare, cooler…), wind effects in the horizontal axis and snow capacity considering the region to be installed.

Figure 3: CASOH pilot plant foundations

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5.1.3 Fluids supply

CASOH pilot plant is an equipment which requires of a high variety of gases and fluids to operate correctly. Moreover, as it is an experimental plant, these utilities must be supplied in the most flexible way, considering a variety of scenarios and gas mixtures. Dedicated gas supplies from the Arcelor Mittal industrial plant were built for the pilot plant.

Due to the high flow rates of gases required for a TRL7 pilot plant, it was maximized the quantity of gases and water that could be supplied from the industrial fluids grid. This enables to have access to the gases from any point in the pilot plant. Considering this, the following gas characteristics were considered for the construction of the pilot plant:

Table 2: Inlet pipeline characteristics of the CASOH pilot plant.

Pipeline	P [barg]	T [°C]	Pipeline DN	Material
Blast Furnace Gas (BFG)	0.08-4,2	40	150	Carbon Steel ASTM scheduled STD
Natural gas	6	25	50	Carbon Steel ASTM scheduled STD
Compressed BFG	8	40	150	Carbon Steel ASTM scheduled STD
Steam	6	165	25	Carbon Steel ASTM scheduled STD
Industrial water	8	25	50	Carbon Steel ASTM scheduled STD
Nitrogen	14	25	50	Carbon Steel ASTM scheduled XS (Heat isolated with 50mm and galvanized plate)

To have availability of the industrial gases in the surrounding of the pilot plant, it was decided to create a fluids ring along the pilot plant site, so any new requirement/modification for CASOH pilot plant can be fulfilled easily.

Blast furnace gas is supplied directly at pressure to the pilot plant. For this, ArcelorMittal uses a compression system that is able to supply BFG process gas up to 4.2 barg of pressure. At the

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entrance of the pilot, the pressure will be modified to the different stages/tests required by the project.

Nitrogen and natural gas are directly supplied from the industrial gas grid. This will enable to provide sufficient inert and fuel gases for the different tests planned. Additionally, this will provide a real scenario to the pilot with the specific characteristics of all the industrial gases.

CASOH pilot plant was connected to this steam network. This steam is supplied by the boilers installed in the steelshop of the Gijón plant and some condition variabilities can occur on the supply. Special attention was put on the condensing temperatures in the pipelines. For this, continuous purging points were installed all along the steam pipelines, to remove as much liquid water as possible.

Figure 4: Utilities ring (left) and industrial gas pipelines connection to laboratory

5.1.4 Electrical works

Electricity for the different equipment of the pilot plant was supplied from the low voltage distribution center. This electricity is supplied by the ArcelorMittal Asturias industrial site. This last point is important to be considered, as the factory is subjected to periods when lack of electrical supply can suddenly occur. This is because ArcelorMittal is a large electrical consumer in the region and to guarantee the stability of the whole electrical network, the factory is vulnerable to this type of power outages. This characteristic proved to be crucial in the HAZID and HAZOP analyses, as the CASOH pilot plant will be required to enter automatically in a safety operation mode at any stage during the operation when/if the power supplies go off, as the time to react to these power outages is very limited.

All the electrical works and calculations were stablished considering the electrical loads of the pilot plant and following the Spanish electrical rules of procedure.

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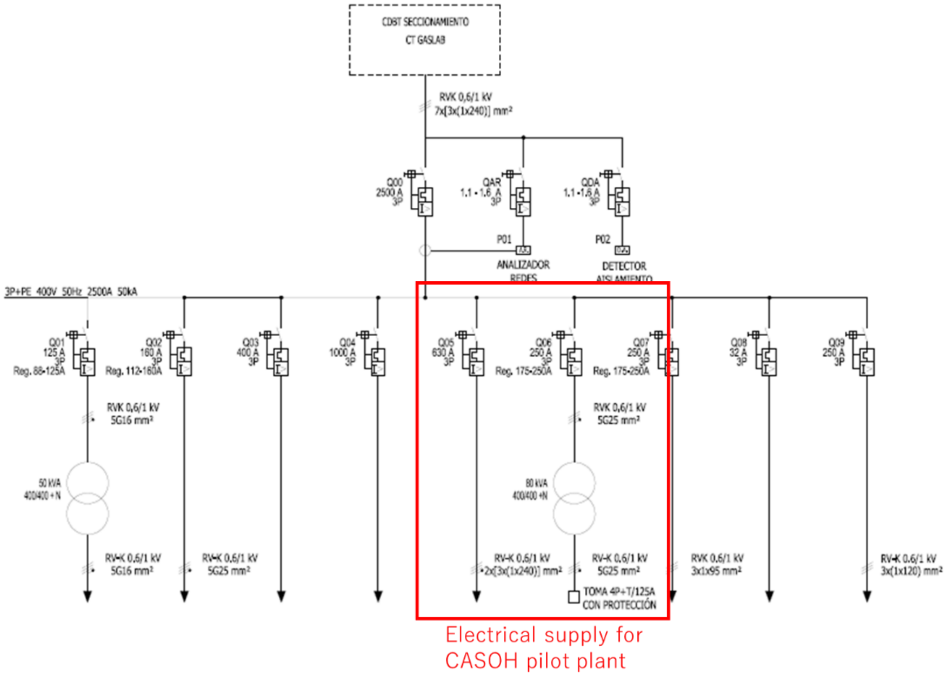

Figure 5: General view on electrical line for CASOH pilot plant

From this electrical supply an electric room was built, in order to store all electrical equipment that can be susceptible to degradation due to weather conditions. From this point, all the electrical connections for the pilot plant were taken.

Figure 6: CASOH electrical room

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5.2 Pilot plant construction

This section describes the pilot plant construction works. Detailed specifications of components are available to project partners at: https://saco.csic.es/index.php/s/YrnExmSW54WPcNd. Such commercial information is mainly proprietary of the suppliers, and will not be included in this section, as such sub-systems are fully manufactured and certified in the premises of the suppliers.

5.2.1 Structure

To conceive the basic structure of the pilot plant, several predesign possibilities were discussed in brain storming sessions. This was done between the technical team of the AMA GasLab and the CSIC team.

The structure of the pilot plant was designed considering the following principles:

Limited footprint: the area for the pilot plant needed to be as compact as possible in terms of surface coverage. This is due to the restrictions in terms of area that the laboratory has due to being inside the industrial factory and the surrounding facilities.

Open to the atmosphere: as this is a gas-based process and due to its size, it is important to reduce as much as possible the risks associated with gas leakages. Having the pilot plant outside will reduce the risks of generating an explosive atmosphere. It is important this this can affect to other equipment installed (weather protections).

Accessibility: as this is a pilot plant, uncertainties about the performance of the different equipment is always a challenge to consider. Accessibility to the reactor is crucial as it is the key element of the pilot plant, and where maintenance manoeuvres are more complex to execute due to the height of equipment.

Taking into count the previous statements, it was decided to build a three-level steel structure at the outside of the laboratory, with the reactor at the most accessible point of the pilot plant (on side). The rest of the equipment were installed strategically at the different heights of the structure.

The total weight of the steel structure accounted for 8387 kgs, including the steel beams, safety fences to comply with regional and on-site regulations, and floor panels.

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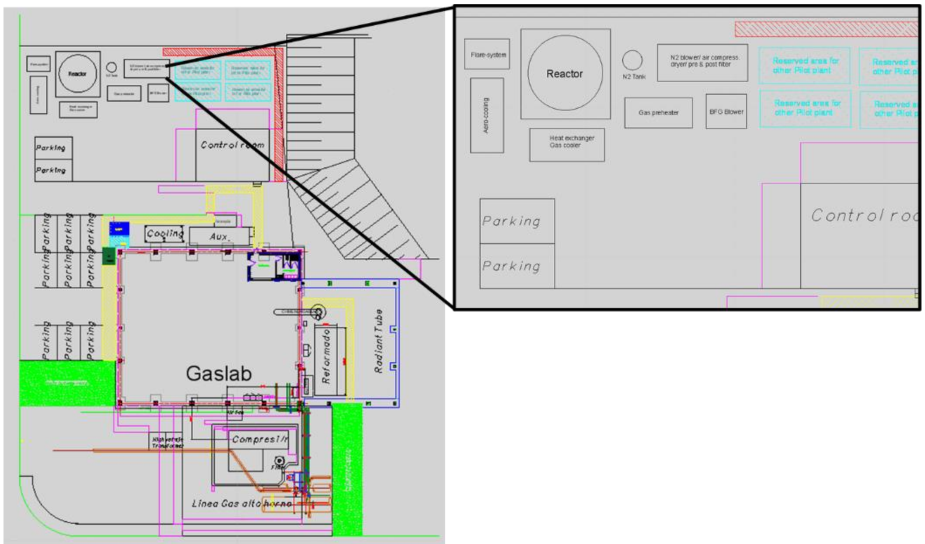

Figure 7: Initial evaluations of the positioning of the pilot plant

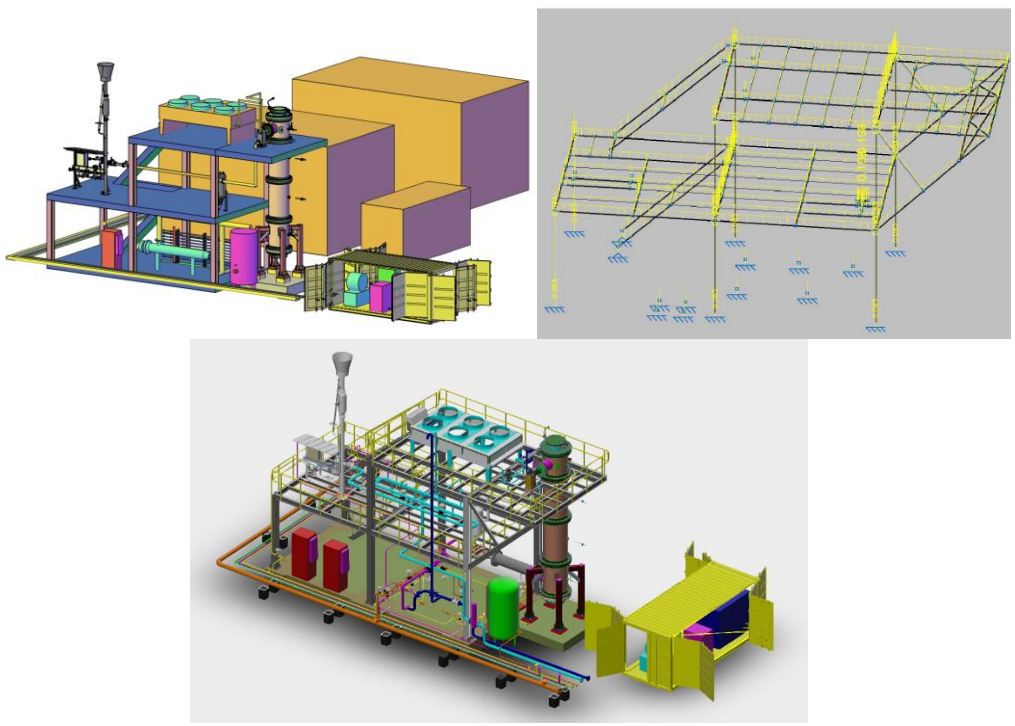

Figure 8: Initial sketches (top-left), structure calculations (top-right) and final 3D scheme (bottom) of the pilot plant.

5.2.2 Reactor construction and installation

As the key equipment of the pilot plant, the design and construction of the reactor was specifically supervised by all the parts involved in the pilot plant construction and operations. These efforts were done as this is the only element in the pilot plant that could not be obtained

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as a standard commercial product. To ensure this, starting from the initial designs of the pilot plant TNO, CSIC and AMIII were involved, with contributions by all partners in WP2 in dedicated meeting for rector design. All the design details of the reactor are included in the D2.2 of the project and no further details will be given in this document.

The full construction of the reactor was done at the in-house facilities of the manufacturing company (Imasa). This ensured that all the quality standards could be done at the same place under controlled conditions and before commissioning at the industrial site. These commissioning tests included:

- Mechanical calculation according to standard EN-13445-3 Ed.2018.
- Equipment pressure fatigue calculation (1 bar to 10 bar for 10,000 cycles @ 100ºC).
- Calculation of pipes to external loads according to the WRC-107/PD-5500 method. If these methods are not applicable, the NozzlePro software was used to perform a simplified finite element calculation to determine the maximum allowable loads in pipes.
- Calculation of support legs.
- Calculation of the reactor to external wind/earthquake loads.
- Calculation of lifting elements (lifting lugs).
- Final equipment drawings (general drawing, details drawing, nozzle drawings and nameplate drawing.

Figure 9: Reactor under construction

According to EN 13445-3 the reactor satisfies the simplified assessment of fatigue life with a design cycles of 10000 for a design temperature and pressure 100ºC and 11barg, respectively.

Additionally, to the construction, the lifting and placement of the reactor on the designated place, was a complex maneuver. Two lifting cranes were used for this task grabbing the reactor by both extremes. After that, the two cranes were coordinated at the same time to place the reactor on the final location. These works were also done with the help of an external company that is dedicated to these works.

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Figure 10: Reactor lifting and placing maneouver

During the material charging procedure executed in August 2023 the interior of the reactor was inspected, not identifying construction damages inside the reactor.

Figure 11: Reactor interior status.

5.2.3 Piping, actuators, and instrumentation.

All the pipe works done in the pilot plant were done following the same directive as in the industrial plant. The initial stages of design were focused on the final definition of the process and instrumentation diagram of the pilot plant (including all the corrections from the different HAZOP analyses carried out by WP2 partners led by TNO, and reviews from the equipment suppliers). This led to the purchasing of the different actuators and instrumentation of the pilot plant (valves, pressure reducers, flowrate controllers…).

In order to reduce the risks on the pilot plant, all of the actuators were obtained following ATEX directive certified as ATEX II 1/2 G Ex ia/d e [ia] IIC T4 Ga/Gb (gas).

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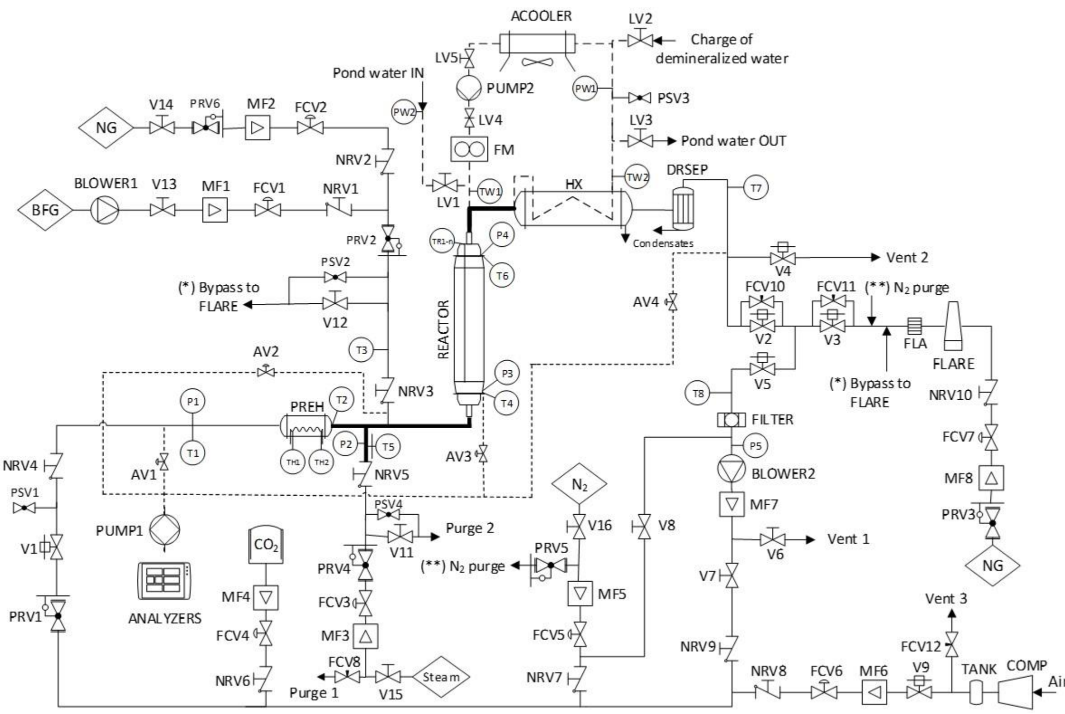

Figure 12: Process flow diagram of the pilot including all actuators as described in D2.2 and Addendum to D2.2

Most of the actuators were supplied by the same supplier, in order to have consistency on all of the equipment before doing the connections with the pipelines. All the piping of the pilot plant was done using carbon steel following the same construction protocols as in the rest of the ArcelorMittal laboratory.

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6 Safety procedures in CASOH pilot plant

Due to the TRL7 scale of the pilot plant, it is necessary to carry out rigorous safety procedures during the construction and operation of the pilot plant. This section compiles all the safety evaluations done in the pilot plant.

HAZID & HAZOP analysis

During the different stages of the design of the pilot plant, several group actions exploiting in the expertise of all WP2 members were taken into consideration. A Hazard Identification Analysis (HAZID) was initially done in the early stages of the pilot plant conception. This workshop targets a qualitative risk for the identification of potential hazards and threats in a process. The purpose is to:

- Review the process at an early stage with a view to ensuring that the process design accounts for credible hazardous scenarios.
- Review safeguards included in the design of the process which are designed to mitigate the relevant risk for the identified hazardous scenarios.

Scope in review		Comments & additional information
Scope/justification		
- Scope and objective identified and understood?		Yes of the porject and of the HAZID
- Business drivers identified?		No relevant
- Risk reduction targets set?		As in sourrounding GasLab premises. AM risk matrix will be used
Project controls		
- Need for hazard studies considered (i.e. HazID, Hazop, Chazop, SIL)?		Yes ,. We will do an initial HAZOP and final HAZOP
- Hazard register reviewed/created?		We will have such list
- Change control process?		No. This is a new process
- Stakeholder involvement/interfaces considered?		All involved
- Contracting strategy?		Following fixed contracting rules at AM and CSIC
- Scope split		No relevant
- Project additionals (technical needs)		Not applicable
Hazard Identification		
- Maintenance hazards		Yes, we will consider this during HAZOP and engineering: in particular operations for solid discharge/charge, cleaning of HX, purging of pilot before opening etc
- Maintenance philosophy identified?		Mantainance procedures of AM will be followed, using specifications from equipment suppliers
- Heavy lifting requirements?		Head of reactor and pieces/section of HX . Full distmantling of reactor. Filling up of solids from the top.
- Access requirements?		Yes, two floor level except to access top of reactor and HX head at apporx 8 m, and instruments in one side of the reactor. The other side of the reactor (without instruments) will have no structure.
- Protection/interlock override for maintenance?		Follow AM rules for mantainance
- Isolation requirements?		No, sourrounded by other pilot facilites
- Confined spaces?		N2 blower and electric heater may be in confined spaces (inside "ship container")
- Services		N2 is a hazard gas in closed environments (N2 blower in container??). CO2 supply probably Dewar type (requires dispersion of CO2 evaporated), BFG services, water, electricity, NG supply.
- Fire water systems?		Fire prevention system of AM will be in place. Fire fighting team in place (AM) in due time will get instructions on the pilot
- Chemical/ fuel storage?		CO2 deward storage. Container(or AM room) for solid storage will be in place. Get data sheets fo materials
- Power supply?		No special needs. Power requirements within AM capabilities on site.

Figure 13: HAZID analysis for the pilot plant (see D2.2 and its annex for more details)

Once the initial design of the pilot plant was done, the HAZOP analysis were performed by WP2 members, led by TNO. It was initially stablished a HAZOP considering the initial flowsheet of the process. In particular, it was used to identify potential hazards in CASOH pilot plant and to identify the operability problems. For that, any change different form the initial was re-´considered.

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DEFINED NODES						
				DRAWINGS		
Node	Description	Stream		Drawing number	Revision	Description
#2 Node 1	Oxidation stage			Fig 1		See Tab 'P&ID for HAZC
#2 Node 2	CASOH			Fig 2		See Tab 'P&ID for HAZC
#2 Node 3	Reduction			Fig 3		See Tab 'P&ID for HAZC
#2 Node 4	Regeneration with BFG			Fig 4		See Tab 'P&ID for HAZC
#2 Node 5	Regeneration with NG			Fig 5		See Tab 'P&ID for HAZC
#2 Node 6	Recarbonation with BFG			Fig 6		See Tab 'P&ID for HAZC
#2 Node 7	Recarbonation with CO2 (mixed with air)			Fig 7		See Tab 'P&ID for HAZC
#2 Node 8	Recarbonation with CO2 and N2			Fig 8		See Tab 'P&ID for HAZC
#2 Node 9	Recarbonation with CO2 and recycle N2			Fig 9		See Tab 'P&ID for HAZC
#2 Node 10	Reduction and carbonisation with BFG			Fig 10		See Tab 'P&ID for HAZC
#2 Node 11	Heating up			Fig 11		See Tab 'P&ID for HAZC
#2 Node 12	Start-up with Air and CO2			Fig 12		See Tab 'P&ID for HAZC
#2 Node 13	Start-up with CO2 and N2 in recycle			Fig 13		See Tab 'P&ID for HAZC
#2 Node 14	Purge with N2			Fig 14		See Tab 'P&ID for HAZC
#2 Node 15	Heat removal after CASOH			Fig 15		See Tab 'P&ID for HAZC
#2 Node 16	Purge with N2 and CO2			Fig 16		See Tab 'P&ID for HAZC
#2 Node 17	Heat removal after regeneration			Fig 17		See Tab 'P&ID for HAZC
#2 Node 18	Safe mode			Fig 18		See Tab 'P&ID for HAZC
	Node finished					
	Node te be reconsidered					
	Node not completed					

Figure 14: Different nodes studied during the HAZOP analysis

Finally, and due to the new process variants considered for CASOH technology ("Enhanced CASOH"), two new operating modes were included in the final HAZOP of the pilot plant. Additionally, **any other change that would occur during the experimental campaign will be evaluated to define if it is necessary or not to do a new evaluation of the HAZOP, that is therefore considered as is a living document**.

Internal safety audits during construction

Once the design phase of the pilot plant was concluded, the construction of the pilot plant commenced. During that activities, internal safety audits were done to the pilot plant in order to detect in an early stage of the construction, different points of improving in the pilot plant.

These activities were also supported by an independent engineering company that also develops commercial systems. By doing it in this way, all the requirements of an industrial plant are also required for the CASOH pilot plant.

These audits consisted in internal meeting explaining in the detail the operation of the pilot. After that, physical revision of the pilot plant was done to see in detail possible improvements in the plant. These improvements will be necessary to solved before the final external audit of the pilot plant (described in the following sections).

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NO CONFORMIDADES Y ACCIONES CORRECTORAS PROPUESTAS

ID	NO CONFORMIDAD	P	I	D	PID	RIESGO	ACCION CORRECTIVA / PREVENTIVA	TIPOLOGIA	COMENTARIOS
ATR0018A-001	Arcelormittal es el integrador de la planta piloto debido a que ha contratado las diversas partidas por separado que componen la planta final. Los contratos lanzados son: - Proyecto de ingeniería (TNO, Impexmón, INCAR, AM) - Construcción y montaje mecánico del reactor (Imasa) y montaje de estructura (Moncobra) - Ingeniería y montaje eléctrico y control (P&D) - Construcción y montaje mecánico de tuberías (Impexmon). - Compra y uso de equipos varios (compresor Aerzen, compresión de gas de cok). El resultado final de la partición de la planta en diversos contratos es que el integrador final es Arcelormittal, y por tanto se debe construir un expediente técnico que contenga toda la documentación técnica relacionada con el diseño, construcción y montaje de la planta. Asimismo, deben realizarse todos los proyectos requeridos por normativa que le afecten.	5	5	5	125	MODERADO	Para formalizar los aspectos legales de la planta en su conjunto es necesario realizar un expediente técnico. Para esto se recomienda: 1- Realizar una evaluación del riesgo de la instalación (ya está realizado por INCAR & AM y plasmado en el informe HAZOP). Sería conveniente una vez instalada la planta volver a revisarla y realizar un segundo análisis de riesgos. 2- Una vez analizados los riegos, de deben implementar las acciones derivadas del informe HAZOP (análisis de riesgos). 3- Realizar los proyectos requeridos por normativa: a. Realizar proyecto de baja tensión (potencia > 20 kW). Se recomienda englobar en un mismo proyecto o como una ampliación del proyecto del cuadro general existente de TSK que englobe las alimentaciones de todas las plantas piloto que se han ido añadiendo: tubos radiantes, reformado de gases, CASOH, Sekisui, compresión de gas de Cok). Si se engloban todas las alimentaciones en un solo proyecto se evita tener que realizar diversos proyectos por separado y es igualmente válido. b. Realizar un proyecto de equipos a gas según RD 919/2006 Reglamento técnico de distribución y utilización de combustibles gaseosos y sus instrucciones técnicas complementarias ICG01 a 11. Es necesario realizar este proyecto debido a que la potencia térmica instalada en la planta supera los 70 kW (se especifican 1000 kW para esta planta). 4- Generación del expediente técnico (parte de esta información está contenida ya en el proyecto de aparato de gas), el cual debe constar de: a. Descripción general del equipo. b. Planos de conjunto de la máquina y planos del circuito de mando (explicaciones pertinentes que se necesiten). c. Documentación relativa a la evaluación de los riesgos (una lista de los requisitos esenciales aplicados y una descripción de las medidas preventivas adoptadas para eliminar los peligros identificados, indicando los riesgos residuales asociados a la máquina) d. Normas y demás explicaciones técnicas utilizadas (ya incluido en el proyecto de Intelmec). e. Cálculos justificativos. f. Informes técnicos que reflejen los resultados de ensayos o pruebas realizadas a los equipos (inspección de soldaduras, certificado de materiales, certificado de soldadores…etc.). g. Manual de instrucciones de la planta (operación, mantenimiento e inspección). h. Sendas copias de la declaración CE de conformidad de los equipos u otros productos incorporados (compresores, válvulas, caudalímetros, equipos de seguridad, aparamenta eléctrica…etc.). i. Copia de la declaración CE de conformidad (automarcado por Arcelormittal en este caso). 5- La planta de compresión de gas de cok debe contemplarse y estar integrada en estos proyectos ya que forma parte de la instalación (etapa de compresión de gas). Se trata de una máquina con su marcado CE que se incorpora al resto de equipos de la planta CASOH. Además, por tratarse de una máquina (le aplica la directiva de seguridad de máquinas), el resto de la instalación se considera también máquina (se junta una máquina con instalación, por tanto además de los proyectos anteriormente citados, la instalación al completo deberá cumplir el RD de seguridad en máquinas).	Legal	La documentación técnica debe exigirse a todos los contratistas de modo que Arcelormittal pueda construir su propio expediente técnico.
ATR0018A-002	Estructura soporte planta piloto: - Falta de drenaje en las patas de la estructura (zona de las cartelas se acumula el agua y puede llegar con el tiempo a corroer la estructura). - Estructura con la toma de tierra aun sin conectar (en fase de montaje).	4	5	4	80	MENOR	- Instalar toma de tierra y conectarla a la red de tierra (ya sale el cable de cobre de la cimentación, falta conexión). Aplicar lo mismo con la estructura del reactor. - Perforar un agujero en las cartelas de los pilares de la estructura para drenar el agua estancada.	Diseño	Se recomienda una sección mínima de cable de puesta a tierra de 50 mm2.
ATR0018A-003	Cordón de soldadura del reactor (parte circunferencial inferior) sin sanear. Toda la soldadura está repasada menos en una zona.	3	3	5	45	MENOR	Solicitar a Imasa que sanee el cordón al completo.	Diseño	
ATR0018A-004	Bandejas de cables aun sin línea de tierra estructural	5	7	5	175	MODERADO	Se recomienda instalar cable de cobre desnudo a lo largo de las bandejas de cable (cuando se ejecute la parte eléctrica).	Diseño	Sección recomendada 50 mm2.
ATR0018A-005	No hay latiguillo de unión entre bridas para garantizar conductividad eléctrica s/ norma interna de ingeniería EGT18.	4	8	5	160	MODERADO	Se recomienda poner a tierra uniendo los tramos de tubería mediante latiguillos de tierra, pletina o malla de cobre (se evita de esta manera la electricidad estática generada en las tuberías por movimiento del fluido en el interior).	Diseño	Se recomienda instalar cable de cobre de sección comprendida entre 6 y 25 mm2.
ATR0018A-006	El aerorefrigerador situado en la parte superior de la estructura para la refrigeración del agua del intercambiador agua-gas no dispone de seta de emergencia, contactores ni relé de seguridad. Por consiguiente, el equipo no está protegido contra arranques intempestivos ni dispone de parada de seguridad.	5	6	5	150	MODERADO	Solicitar la instalación de una seta de emergencia en el propio cuadro, y por consiguiente contactores y relé de seguridad. Además, los contactores podrán usarse para implementar una parada de emergencia general de la planta desde el control.	Diseño	
ATR0018A-007	La chapa del marcado CE del reactor (IMASA) es irregular, ya que el símbolo de marcado CE no está normalizado.	3	3	3	27	MENOR	Solicitar a IMASA una placa de marcado CE nueva que cumpla la normativa de marcado.	Legal	
ATR0018A-008	Tornillos de unión de tramos de tubería no sobresalen a penas nada de las tuercas.	5	7	5	175	MODERADO	Tener en cuenta a la hora del montaje el uso de tornillos de la longitud adecuada.	Diseño	Se recomienda que las roscas de los tornillos sobresalgan al menos 5 mm de las tuercas para que estos trabajen de la forma prevista.
ATR0018A-009	Cartelería de riesgos en inglés en el compresor Aerzen	3	3	3	27	MENOR	Traducir cartelería e identificación de botones y riesgos al español.	H&S	
ATR0018A-010	Zona de poleas del compresor de tornillo para el N2 del compresor Aerzen es accesible. (zona del eje).	3	9	4	108	MODERADO	Falta protección en la zona del eje. Parece estar preparada para poner una chapa que está ausente. Buscar chapa o instalar una nueva.	H&S	
ATR0017A-011	No se ha realizado ningún estudio ATEX en base al diseño la planta.	3	9	6	162	MODERADO	En base al layout de tuberías y demás elementos, se recomienda realizar un estudio ATEX para prevenir la generación de atmosferas explosivas en la zona. Del estudio podrán derivar acciones que modifiquen la implantación diseñada o bien se requiera equipos con especificación ATEX.	H&S	Siempre que se realice una planta piloto con gases de proceso combustibles, se recomienda lanzar previamente un estudio ATEX en base al layout, de forma que se puedan prever modificaciones en el diseño inicial antes de la construcción de la planta. En este caso particular, al encontrarse la planta a la intemperie la peligrosidad se reduce respecto a una instalación en interior, pero de igual modo se debería revisar.

Figure 15: Points of improved after internal evaluations of the pilot plant during the construction

*This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Grant Agreement No **884418***

Figure 16: Images for points of improvement in the pilot plant

ATEX study

Due to the presence of fuel gases in the pilot plant, it was necessary to perform an initial ATEX evaluation of the facility in order to detect possible improvement points during the design and construction of the pilot plant. This would help to avoid future modifications due to the necessity to fulfil with ATEX regulation.

The study verified the compliance and adequacy of the facilities with Royal Decree 681/2003 on the protection and safety of workers exposed to risks derived from explosive atmospheres in the workplace.

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ARCELORMITTAL ESPAÑA - PLANTA PILOTO (Rev.1 Octubre 2021)
(clasificación realizada según la norma UNE-EN 60079-10-1:2016)

FUENTE DE ESCAPE					SUSTANCIA INFLAMABLE				VENTILACIÓN			EMPLAZAMIENTO PELIGROSO				
Descripción	Ubicación	Grado de escape [a]	Tasa de escape (kg/s)	Características del escape (m^3/s)	Temperatura y presión de servicio		Sustancia	Estado [b]	Tipo [c]	Grado de dilución	Disponibilidad	Tipo de zona 0-1-2 [d]	Extensión de la zona (m)		Observaciones	MARCADO DE EQUIPOS
					°C	bar							V	H		
Bomba	BFG feed	S	3,53E-03	2,10E-02	25	2	GHA	G	N	Medio	Justa	2	0,8	0,8	Esfera de radio 0,8 metros Tubería diámetro 75 mm Sección de fuga 5 mm2 Altura < 2 m	II 3G EEx IIA T1
Picajes de medida / Válvula de corte / Válvula antiretorno / Brida / Brida ciega / Accesorio de tubería	BFG feed	S	1,77E-03	1,10E-02	25	2	GHA	G	N	Medio	Justa	2	0,6	0,6	Esfera de radio 0,6 metros Tubería diámetro 75 mm Sección de fuga 2,5 mm2 Altura < 2 m	
Picajes de medida / Válvula de corte / Válvula antiretorno / Brida / Brida ciega / Accesorio de tubería	NG feed	S	5,53E-04	3,20E-02	25	2	GN	G	N	Medio	Justa	2	0,4	0,4	Esfera de radio 0,4 metros Tubería diámetro 25 mm Sección de fuga 1 mm2 Altura < 2 m	
Picajes de medida / Válvula de corte / Válvula antiretorno / Brida / Brida ciega / Accesorio de tubería	Reactor feed	S	5,97E-04	7,90E-02	500	2	GCK	G	N	Medio	Justa	2	1,5	1,5	Esfera de radio 1,5 metro Tubería diámetro 100 mm Sección de fuga 2,5 mm2 Altura < 2 m	II 3G EEx IIB T1
Válvula de seguridad al exterior	Reactor	S	7,96E-04	1,05E-01	500	3	GCK	G	N	Medio	Justa	2	1,5	1,5	Esfera de radio 1,5 metros Diámetro válvula 25 mm Sección de fuga 0,1 x sección orificio Tubería diámetro 100 mm Altura < 2 m	
Picajes de medida / Brida / Brida ciega / Accesorio de tubería	Reactor out	S	3,38E-05	4,00E-03	870	Atmosférica	GCK	G	N	Alto	Justa	2ED	-	-	Presión: 0,01 bar Tubería diámetro 168 mm Sección de fuga 2,5 mm2 Altura > 5 m	No Aplica
Picajes de medida / Brida / Brida ciega / Accesorio de tubería	Heat Exchanger	S	3,38E-05	4,00E-03	870	Atmosférica	GCK	G	N	Alto	Justa	2ED	-	-	Presión: 0,01 bar Tubería diámetro 125 mm Sección de fuga 2,5 mm2 Altura > 5 m	
Picajes de medida / Válvula de corte / Válvula de control de flujo / Válvula de purga / Brida / Brida ciega / Accesorio de tubería	Downstream Heat Exch.	S	6,26E-05	8,00E-03	60	Atmosférica	GCK	G	N	Alto	Justa	2ED	-	-	Presión: 0,01 bar Tubería diámetro 100 mm Sección de fuga 2,5 mm2 Altura > 2 m hasta 5 m	
Válvula de control de flujo / Válvula antiretorno / Brida / Brida ciega / Accesorio de tubería	NG to Torch	S	5,53E-04	3,20E-02	25	2	GN	G	N	Medio	Justa	2	0,4	0,4	Esfera de radio 0,4 metros Tubería diámetro 25 mm Sección de fuga 1 mm2 Altura < 2 m	II 3G EEx IIA T1
Válvula / Válvula de corte / Brida / Brida ciega / Accesorio de tubería	Gas analysis	S	8,71E-06	1,00E-03	100	Atmosférica	GN	G	N	Alto	Justa	2ED	-	-	Presión: 0,01 bar Tubería diámetro 6 mm Sección de fuga 0,25 mm2 Altura < 2 m	No Aplica
Bomba	Gas analysis	S	3,49E-05	2,00E-03	100	Atmosférica	GN	G	N	Alto	Justa	2ED	-	-	Presión: 0,01 bar Tubería diámetro 6 mm Sección de fuga 1 mm2 Altura < 2 m	

[a] Grado de escape: C-Continuo; S-Secundario; P-Primario
[b] Estado: G-Gas; L-Líquido; LG-Gas licuado; S-Sólido
[c] Tipo: N-natural; AG-General artificial; AL-Local artificial
[d] Tipo de zona peligrosa: zona 0ED, 1ED, 2ED, indica una zona teórica despreciable en condiciones normales.

Figure 17: Definition of safety distances in the evaluation of potential explosive areas.

During the evaluation, one of the major risks detected was linked with the pressure safety valve of the reactor of the pilot plant. As this is a valve that can suddenly be opened by an excess of pressure in the reactor, the safety sphere calculated for this valve was 1.5 m in diameter.

For this reason, it was decided to move the non ATEX equipment (gas preheaters) to an area outside the safety sphere. This was an important modification on the pilot plant that was assessed in an early stage to mitigate further delays during the construction.

The rest of the safety spheres in the pilot plant are much smaller (<1 m) and due to the acquisition of ATEX instrumentation it was not necessary to change drastically the initial design of the pilot plant as described in Deliverable 2.2.

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Safety protocols during the construction:

In this section of the document, it is necessary to mention that during the execution of the construction of the pilot plant, all the safety considerations that are applicable to any industrial site of ArcelorMittal where implemented:

1) Prior to any work in the facilities, it was necessary to sign a contract between the company supplying the construction service and ArcelorMittal. The signature of a contract requires the update of all the safety documentation of every company in an internal platform that checks that everything is correct.
2) Once the company is accepted, all the workers have to pass an exam where all the internal safety obligations are ensured that are known by any worker. Specifically, at Gaslab (R&D lab) a safety training course is done the first day in order to highlight the specific risks of the laboratory.
3) On weekly basis every company has to fill in the General Safety Order (OGS), where the risks of the facility are reviewed personally by each company.
4) Finally, on daily basis, all the companies that are willing to execute any work during the day in the laboratory have to attend to the initial meeting (first hour in the morning) to describe the works to be performed. Additionally, the Hira Lite procedure is also done in order to review the proper risks of the work that is going to be done that day and if there are some companies working in the surroundings a coordination protocol is also done.

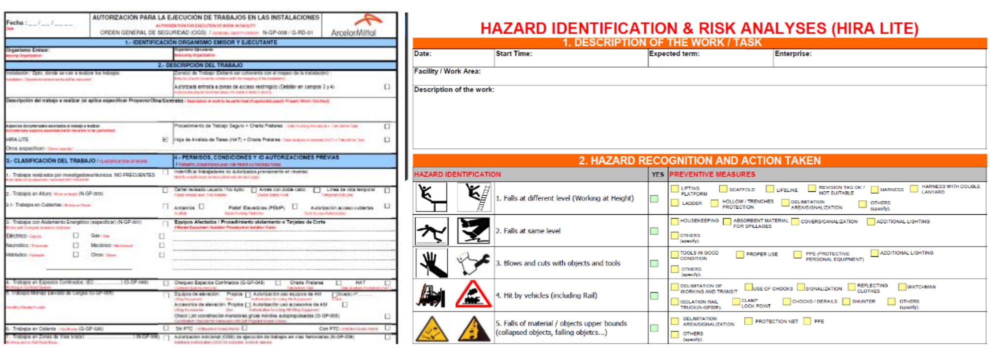

Figure 18: OGS and Hira lite protocols

During all the construction of the pilot plant, all the accesses to the pilot plant were delimited by a fence. Any worker not involved in the project was not allowed to enter the pilot plant. Additionally, any worker without the convenient PPEs was not allowed to access the pilot plant area.

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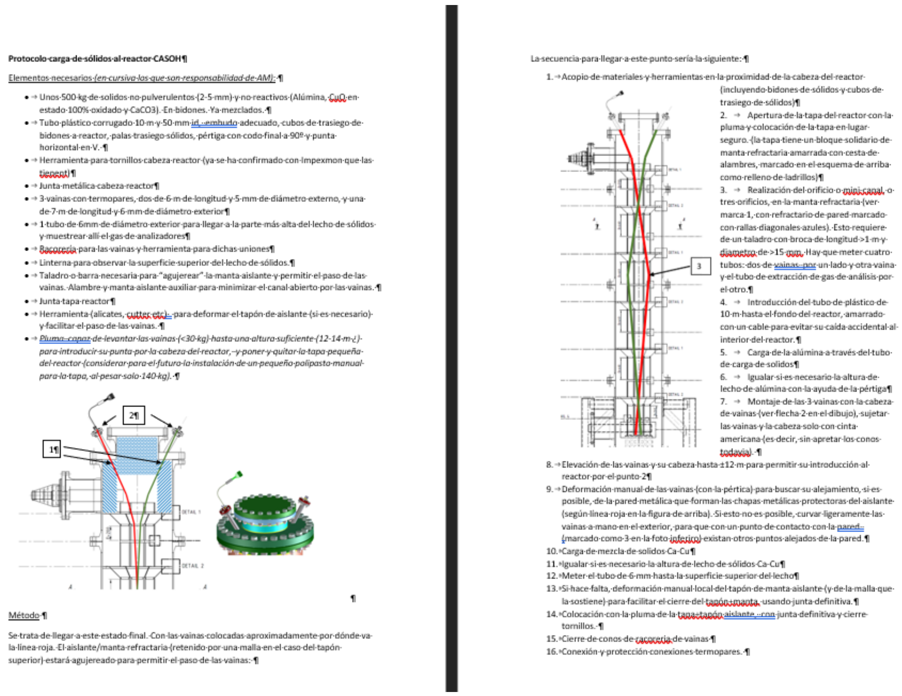
7 Commissioning tests

Once that the pilot was assembled, and in order to get the final validation before introducing any type of fuel gas in the system to carry out a CASOH experiments, it is necessary to ensure the correct control of the entire plant. To do this, a sequence of up-scaling test has been done to ensure that the operation of the pilot plant is ensured in the most appropriate and safe way.

All of these commissioning tests were done using nitrogen as operating gas, in order to mitigate risks.

7.1 Material loading to the reactor.

To fill the reactor with the chosen functional material mixture of Cu-catalyst supplied by Johnson Matthey and Ca-material supplied by Carmeuse (see D2.2 and D2.3), it was necessary to stablish a safety procedure, as this is an operation that involves the moving of heavy and suspended loads. This procedure has been approved by the different companies involved in the operation (AM, CSIC and mechanical mounting service provider). These and other safety documents are compiled in Spanish to fulfil with the legal requirements.



Protocolo carga de sólidos al reactor CASOH

Elementos necesarios *(en cursiva los que son responsabilidad de AM)*:

- Unos 500 kg de solidos no pulverulentos (2-5 mm) y no reactivos (Alúmina, CuO en estado 100% oxidado y CaCO3). En bidones. Ya mezclados.
- Tubo plástico corrugado 10 m y 50 mm id, embudo adecuado, cubos de trasiego de bidones a reactor, palas trasiego sólidos, pértiga con codo final a 90º y punta horizontal en V.
- Herramienta para tornillos cabeza reactor (ya se ha confirmado con Impexmon que las tienen!)
- Junta metálica cabeza reactor
- 3 vainas con termopares, dos de 6 m de longitud y 5 mm de diámetro externo, y una de 7 m de longitud y 6 mm de diámetro exterior
- 1 tubo de 6mm de diámetro exterior para llegar a la parte más alta del lecho de sólidos y muestrear allí el gas de analizadores
- Racorería para las vainas y herramienta para dichas uniones
- Linterna para observar la superficie superior del lecho de sólidos.
- Taladro o barra necesaria para "agujerear" la manta aislante y permitir el paso de las vainas. Alambre y manta aislante auxiliar para minimizar el canal abierto por las vainas.
- Junta tapa reactor
- Herramienta (alicates, cutter etc). para deformar el tapón de aislante (si es necesario) y facilitar el paso de las vainas.
- *Pluma capaz de levantar las vainas (<30 kg) hasta una altura suficiente (12-14 m ¿) para introducir su punta por la cabeza del reactor, y poner y quitar la tapa pequeña del reactor (considerar para el futuro la instalación de un pequeño polipasto manual para la tapa, al pesar solo 140 kg).*

Método

Se trata de llegar a este estado final. Con las vainas colocadas aproximadamente por dónde va la linea roja. El aislante/manta refractaria (retenido por una malla en el caso del tapón superior) estará agujereado para permitir el paso de las vainas:

La secuencia para llegar a este punto sería la siguiente:

1. Acopio de materiales y herramientas en la proximidad de la cabeza del reactor (incluyendo bidones de sólidos y cubos de trasiego de sólidos)
2. Apertura de la tapa del reactor con la pluma y colocación de la tapa en lugar seguro. (la tapa tiene un bloque solidario de manta refractaria amarrada con cesta de alambres, marcado en el esquema de arriba como relleno de ladrillos)
3. Realización del orificio o mini canal, o tres orificios, en la manta refractaria (ver marca 1, con refractario de pared marcado con rallas diagonales azules). Esto requiere de un taladro con broca de longitud >1 m y diametro de >15 mm. Hay que meter cuatro tubos: dos de vainas por un lado y otra vaina y el tubo de extracción de gas de análisis por el otro.
4. Introducción del tubo de plástico de 10 m hasta el fondo del reactor, amarrado con un cable para evitar su caida accidental al interior del reactor.
5. Carga de la alúmina a través del tubo de carga de solidos
6. Igualar si es necesario la altura de lecho de alúmina con la ayuda de la pértiga
7. Montaje de las 3 vainas con la cabeza de vainas (ver flecha 2 en el dibujo), sujetar las vainas y la cabeza solo con cinta americana (es decir, sin apretar los conos todavia).
8. Elevación de las vainas y su cabeza hasta ±12 m para permitir su introducción al reactor por el punto 2
9. Deformación manual de las vainas (con la pértica) para buscar su alejamiento, si es posible, de la pared metálica que forman las chapas metálicas protectoras del aislante (según linea roja en la figura de arriba). Si esto no es posible, curvar ligeramente las vainas a mano en el exterior, para que con un punto de contacto con la pared (marcado como 3 en la foto inferiro) existan otros puntos alejados de la pared.
10. Carga de mezcla de solidos Ca-Cu
11. Igualar si es necesario la altura de lecho de sólidos Ca-Cu
12. Meter el tubo de 6 mm hasta la superficie superior del lecho
13. Si hace falta, deformación manual local del tapón de manta aislante (y de la malla que la sostiene) para facilitar el cierre del tapón+manta, usando junta definitiva.
14. Colocación con la pluma de la tapa+tapón aislante, con junta definitiva y cierre tornillos.
15. Cierre de conos de racoreria de vainas
16. Conexión y protección conexiones termopares.

Figure 19: Solids load protocol inside the reactor

The reactor was filled with three different types of materials. From bottom to top they are described as follows:

Alumina pellets (50 liters): before placing any type of functional material in the reactor, it is necessary to leave some inert space between the gas distributor of the reactor and the

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beginning of the bed of solids to be studied. In this case alumina round pellets were selected for this purpose. Alumina is an inert material with high heat capacity that is used for two purposes: distribute evenly the gas flow along the bed of solids and homogenize the temperature of the inlet gas prior to any contact with the functional materials.

Functional materials, copper and calcium carbonate pellets (200kg): This material mixture was prepared before the reactor loading. This is necessary as these materials have to be into close contact between each other and homogenously distributed along the different heights of the bed of solids. This is due to the exothermic reactions that occur during the CASOH reactions. If the material is not perfectly homogenized, some hot spots could be created in the bed of solids. These hot spots could lead to some viscous state of the metallic copper pellets leading to partial clogging of the bed of solids. In order to maintain the integrity of the functional materials, a corrugated pipeline was used to reduce the falling force and reduce the dust formation at the bottom of the reactor due to loading.

The total height of the solids currently is 1 m in order to tests the performance of the reactions. Once these first experimental tests are executed, more material is planned to be loaded inside to increase the production capacity of the reactor.

Figure 20: Material loading in the reactor (left and middle) and reactor material structure (right)

7.2 Piping final works: cleaning and utilities supply

Piping cleaning is an important task in the acceptance tests as it prevents any type of future damage or blockage in the pilot plant or its sensors, due to the presence of metallic parts that can be inside due to grinding and welding works in the pipelines.

These cleaning works were done by isolating different sections of the pilot plant. Initially compressed air was used to blow manually the bigger parts of the pipelines until no dust could be seen. These tests were useful to make some slight modifications to the gas lines as other purging connections were done at the pilot plant.

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Once this initial cleaning was done, it was necessary to confirm the availability of the different utilities to be supplied to the pilot plant. For that, the different upstream actuators were opened, and it was visually inspected that the utilities were at the inlet valves of pilot plant.

Nitrogen: nitrogen from the AMA factory gas network was checked, and pressure at the entrance of the pilot plant was at 14 barg. Two pressure reducing valves were installed. One was used to supply N2 to the control circuit of the different plants and the other one for the inlet gas for purging and cooling the pilot plant (process gas). The pressure of these two lines was selected to 8 and 3 barg respectively.

Blast Furnace Gas: blast furnace gas was also checked in one of the venting connections of the general pipeline. This was inspected visually as well as with gas detectors that are capable to measure CO concentration. The compression system was also checked during the last months and being capable to supply BFG at pressures up to 4.2 barg.

Figure 21: Compression plant and internals modified to operate with BFG

Natural Gas: Natural gas was inspected due to the built-in pressure of the main pipeline. As this is a gas that comes pressurized, it is easy to detect it by the pressure gauges.

Steam: Steam was checked on the just before the reactor entrance to ensure that steam line is supplying it properly. To check the availability of steam, different purging valves were carefully opened in a sequential way and observing the presence of steam along the pipeline.

Water: the supply of industrial water is only on severe emergency case and could not be checked until new automatic valves are installed. This is a pending task to improve the emergency system. The supply of demineralized water was done as the cooling circuit is a closed circuit that has to be charged "manually" using a water pump.

Finally, a second cleaning step was done to ensure that cleanliness of the pipelines is sufficient to carry out the experiments. This second cleaning was done using process nitrogen blow the different sections of the pipelines before the proper closing of all pipe connections. To do this, a flexible hose was connected to introduce nitrogen at different points of the pilot plant, and it

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was visually inspected that no dust was coming out of the outlet pipeline. Once all the pipes were clear a retightening of the pipelines joints was done.

Figure 22: Example of a cleaning test, showing dust and debris coming out from the N2 line to the preheaters.

7.3 Actuators checking

Before introducing any gas in the reactor or any automation activity, it was necessary to test individually any actuators installed in the pilot plant. To ensure this, the same service company in charge of programming and installing the control of the plant, has collaborated to check all the signals (analogic and digital) and status of the pilot plant actuators.

Once all the signals were checked, pneumatic actuators were energized with pressurized nitrogen to check also individually that all the actuators work at ambient pressure (gas circuit still opened to atmosphere. This was an important milestone to achieve in the factory acceptance test, as CASOH is characterized by its conditions swing nature. All the valve systems must work co-ordinately to ensure that all the reactions and safety conditions are ensured during the operation. However, on these tests, 5 valves were found to operate in the opposite way (i.e. they were "normally open" when the pilot was off or in safe mode, when they should be "normally closed" in such operating modes). To solve this issue, the valves were disassembled, and the internals switched to the correct position.

One pending subject emerging from these tests is that PCV1 valve has to be modified to ensure that the valve is "normally closed". This modification will be done once the valve is available to be supplied. This valve is resoundingly working with other 3 valves, that act as safety conditions to operate the pilot plant. Therefore, it is important to remark that this small problem is not an inconvenience to conclude the acceptance tests.

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7.4 Pipeline gas tightness

After checking all the instrumentation, it was necessary to check for any fluid leakage on the plant. In this type of gas-based pilot plants, it is necessary to detect any gas leaks along the different joints of the pipelines. For this purpose, acceptance tests were taken in order to ensure the proper sealing of the facility. Once nitrogen was available on the pilot plant. Different sections of the pilot were filled with N2 to detect any leaks that could be detected visually using soapy water.

These tests have to be done with nitrogen and two different pressures were selected to perform these tests:

- Pressure of 8 barg: selected for the lines that are designed to withstand high pressures in the pilot plant. To reach this pressure it is not necessary for the pilot plant but ensures gas tightness at higher levels.
- 0.5 barg: selected for the preheaters and the reactor. As preheaters are just at the entrance of the pilot and they limit the pressure that can be introduce in the reactor.

Some minor leaks were detected and solved by retightening the bolts of the flanges. In some cases, this was not sufficient, and the gaskets of the pipelines were replaced. In these tests three major points of improvement were detected:

BFG inlet: the connections of the different actuators were not properly tightened and a retightening of this section of the pilot plant was necessary to be done.

NG inlet: The gas conditioning valves could not be checked, as PRV6 valve was preventing the nitrogen to enter in this section of the pilot plant. As the initial tests of CASOH will not require NG, this will not delay the acceptance of the pilot plant even though it will be necessary to done before introducing such gas.

Reactor: it was detected a major leakage in the DN1000 connection of the reactor of the pilot plant. As this is a crucial element, it was decided to focus on solving this problem. Due to the importance of this element a retightening of the entire element will be done by a certified company to ensures that all the bolts are tightened at the proper torque.

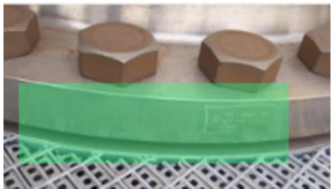

Figure 23: Flange where gas leak was detected in the reactor

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In summary the following lines were tested to be gas tightened:

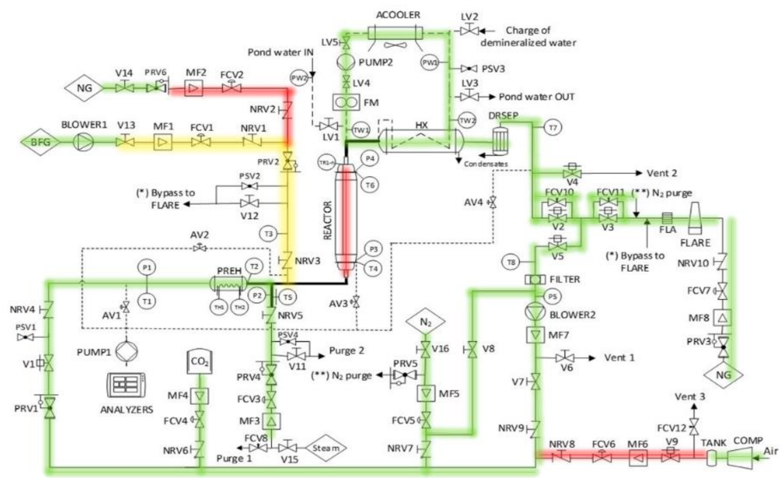

Figure 24: Gas tightness of the pilot plant after acceptance tests.

As can be seen in the image above, there are some lines highlighted in red. To ensure the proper functioning of the pilot plant, these leakages have to be fixed.

As final check, and once the previous points are fixed, the pilot plant will be filled with nitrogen at the correct pressures, and it was left under pressure for 5 hours to ensure that no leaks are present in the pilot plant.

7.5 Off-gas management: cooling and flaring

During the design of the pilot plant, it was decided that the fuel gases that are generated in the pilot plant are flared with an automatic torch system that is able to minimize the consumption of natural gas in the flare. This system controls the amount of NG by the incoming flowrate and the composition of the fuel gases that are necessary to be combusted.

Before the combustion of fuel gases in the torch, it is necessary to cool down the gases. For this purpose, a water-cooled circuit is used. The system was tested to see that all the fans were operating correctly before introducing any hot gas into the pilot plant. Prior to this, water was pumped to the water-cooling circuit and air was purged from the highest purging connection.

During the beginning of acceptance trials, it was detected that the fans of the pilot were not moving. An initial source of the problem could be the PLC and local tests with the supplier were done at their facilities. A new PLC was supplied by the company manufacturing the water cooler, as the problem was not solved, and the source was not detected.

Finally, a shortcut was detected in one of the connections of the fans (some earth derivation as a cable was pinched with an electrical box). This was repaired and the fan system was properly working. These tests were very useful as it is important to notice that if a fan stops

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working the entire air-water cooling system will shut down and the CASOH pilot would enter into safe mode (see D2.2).

Figure 25: Replaced cable for the water-air cooling system

Once the operation tests of the cooling system were successful, the flaring system was checked. To test this system, fuel gases were introduced to the system at a pressure up to 2barg to ensure the proper working.

Figure 26: Off gas system tested with fuel gases during acceptance tests.

The rest of the generated gases in the pilot plant will be either vented or recirculated as in terms of safety are inert compounds.

7.6 Pilot plant control, automation, and safety interlocks

Once the leaking tests were fulfilled and the control of the valves were checked. The control of the pilot plant was monitored. A layout of the pilot plant was represented on a Supervisory Control And Data Acquisition software (SCADA).

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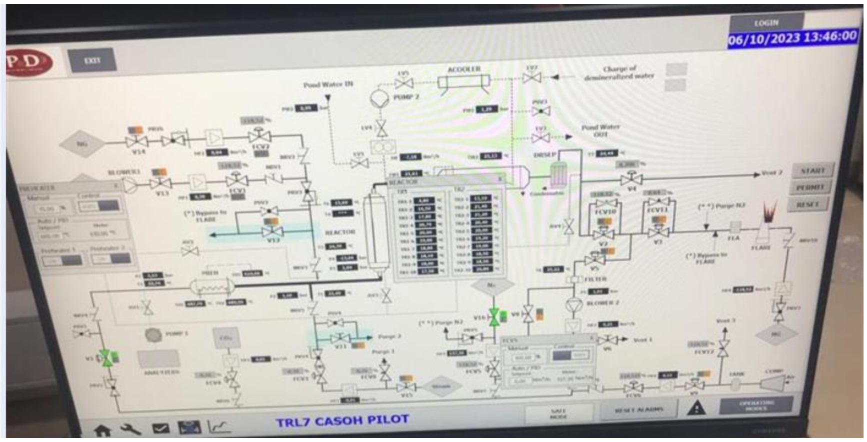

Figure 27: SCADA for CASOH pilot plant

To do this, all the different stages of the system are programmed. The stages will not be described in this document, as they are already described in D2.2 and its Addendum to D2.2. After this, and using an inert atmosphere, the proper actuation of the instruments was checked for the pilot plant. This action was successfully done and the automation of the different stages of CASOH process are being implemented. At this stage it is important to check the safety from 2 different perspective:

- Base safety: Stablish and ensure that the high and low values of each instrumentations are measured correctly. If the values are out of the range to ensure a safe condition for the next process stage the pilot control will not allow the operator to move to the next operation mode. If the values are out of the range for the design of the pilot plant, the pilot will enter automatically in a safe mode, to protect all the equipment.
- Reactive safety: if none of these limits act properly, there are physical safety equipment that prevent the excursion of parameters such as the pressure. All of these systems are necessary to be tested. In particular PSV1 was successfully checked with over pressure at the entrance of the reactor (>1.5 bara).

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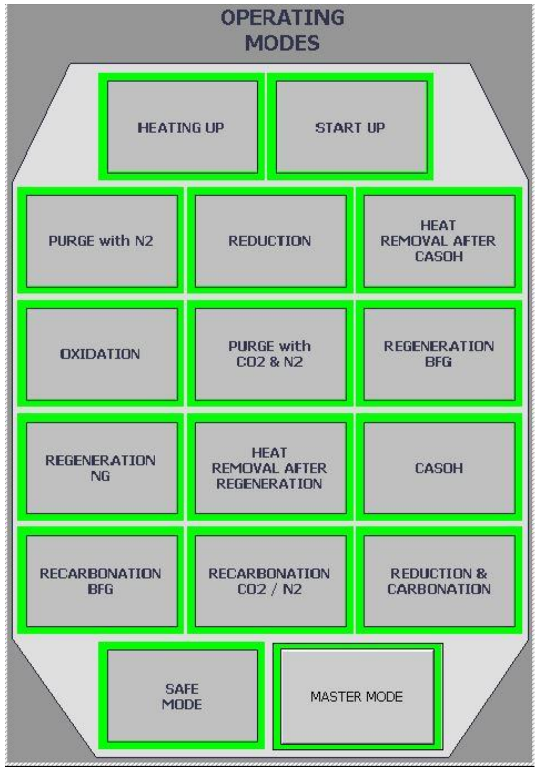

Figure 28: Operating modes programmed in the pilot plant

7.7 Gas heating tests

Once all the previous work was done, and before the final automatic test of the plant, heating tests were conducted as final stage on the acceptance tests of the pilot plant. This was a crucial step on the pilot construction as both preheaters are one of the most sensitive pieces of equipment in the pilot plant. Isolated heating tests were done to determine the performance of the heaters and the impact of the heat in the different sections of the plants.

To do these tests it was necessary to operate the pilot plant with nitrogen as these systems require continuous gas flow to ensure that the elements are not overheated. During these tests the preheaters were set at approximately 600°C to see the evolution of the temperature of the gas small changes in temperature were detected as the power to the preheaters was not set to the maximum in order to extend the lifetime of the equipment. Small heating slopes will preserve to the maximum the lifetime.

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The temperature profiles of these tests are shown in the following graphs:

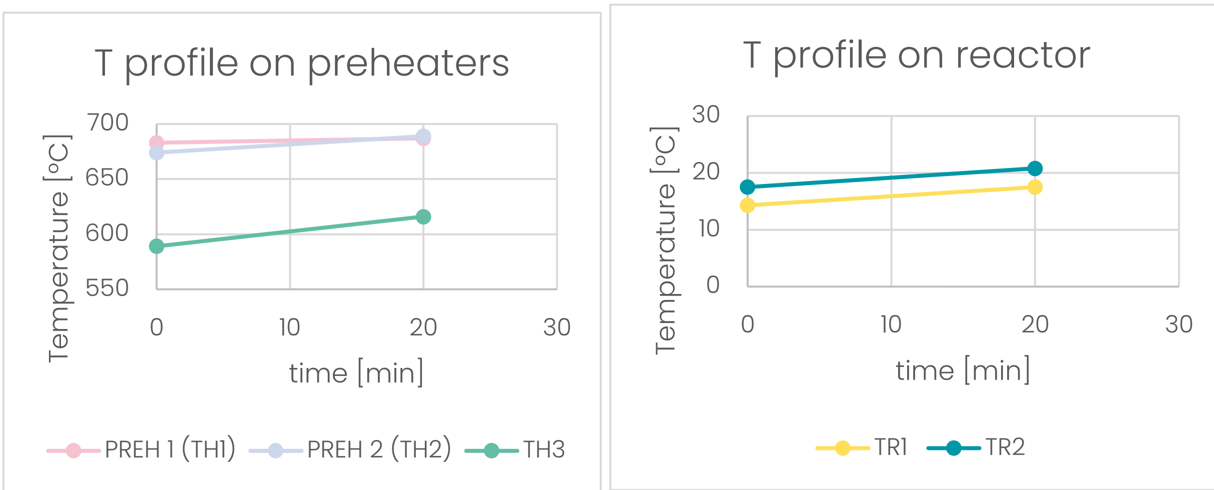

Figure 29: Results of heating in acceptance tests.

As can be seen in previous figures, even though the temperature increase in the nitrogen stream was not very high. It was tested the operating temperature of the preheaters. The preheaters were operating properly at >600ºC with pressures up to 0.5 barg. These conditions showed good performance of the preheaters under realistic operating conditions for the pilot plant.

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7.8 Conclusions and action plan

Final acceptance tests have been conducted to ensure that all the operation of the pilot plant is working properly in terms of operation and safety. These tests were established to determine the operational performance of the plot plant. After the different trials it can be concluded that the physical construction of the pilot plant is finished, but small points of improvements were highlighted before fuel gases can be fed to the pilot. The following table summarized the action points for the pilot plant operators at AMA and CSIC teams:

Table 3: Results of pilot plant acceptance tests

Equipment	Action plan
Terrain and foundation	Finished
Electrical supply	Finished
Gas supply (BFG, Steam, N2, NG, Air)	Finished
Structure	Minor details (rainwater accumulation, paint, cleanliness…) to be solved in parallel to the tests.
Piping	Finished (small improvements are expected to be done during execution of experiments, such as extra connections or purging outlets.).
Instrumentation	Finished but it will be necessary to re-calibrate just before trials with BFG
Actuators	To replace some manual valves to automatic in the water-emergency system. PCV10 replacement. Recalibrate all the valves before the tests.
Preheaters	Finished
Reactor	Re-tighten and certify the proper torque of each bolt in the reactor.
Water-Air cooling system	Finished, improve by putting some automatic air purges in the highest pipelines.
Drop separator	Finished
Flaring system	Finished
Venting system	Finished

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Automatic control	Safety is being finished – conduct the 2 days automatic test before the trials with flue gases.

After the finalization of the acceptance tests, the action points specified in the document will be done. These actions will be carried out in parallel to the tests stablished in the C^4U project as they are internal and external requirements of ArcelorMittal.

Finally, and due to the entity of the pilot plant, final documentation and safety audits will be done in parallel to the execution of the tests in order to determine and mitigate any risk that could be identified in the future. These actions will include:

- As-built mechanical and electrical documents. These documents will include the changes that are contemplated in this deliverable.
- Final HAZOP revision.
- Adequation to Spanish experimental plant requirements (RD1215 adequation).
- Final internal safety audit for pilot plants.
- Minor safety changes in the pilot plant.
- Final external audit of the pilot plant (certified company).

All of this information will be compiled between ArcelorMittal laboratory and CSIC-INCAR complying with the internal and external requirements for the CASOH pilot plant facility.

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