

Combustion of Rechargeable Metal Fuels in Fluidized Bed Boilers

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Results of medium scale fluidized bed tests

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Author(s): M. de las Obras Loscertales, A. Abad

Affiliation: Agencia Estatal Consejo Superior de Investigaciones Científicas (CSIC)



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1 Introduction

The overall objective of the CORAL project is to facilitate the transition toward a more sustainable energy system by replacing fossil fuels with renewable energy sources. In this context, the project aims to demonstrate the feasibility of a thermochemical energy storage system based on metal oxides — particularly iron oxide— by retrofitting existing coal-fired fluidized bed boilers.

As an initial step, small-scale laboratory experiments (D2.1) were conducted. Among several potential candidates, the material exhibiting the most promising performance, Ferelet MT, was selected for further investigation in a medium-scale fluidized bed reactor, which is the focus of this deliverable. The results obtained herein are expected to play a key role in the subsequent demonstration of the process in a 1 MWth circulating fluidized bed pilot plant (TRL 6 in Task 2.4), as well as in the validation of the fluidized bed oxidation models developed within the project (WP3).

The main objective of the medium-scale experimental campaign (T2.2) is to identify the feasible operating window of the process. To this end, a series of experiments was conducted in a 3 kWth bubbling fluidized bed reactor. The primary operating variables investigated include temperature, particle mean residence time within the reactor, air ratio, and fluidization gas velocity.

In addition, the tendency of the selected material to undergo agglomeration during the oxidation stage was evaluated in order to prevent defluidization.

Following this introduction, the report presents a detailed description of the experimental facility, along with the commissioning tests carried out. Subsequently, the main operational challenges and the results obtained are discussed, with particular emphasis on the influence of key operating parameters on the oxidation degree of the iron-based material.

2 Medium Scale Fluidized Bed Reactor

2.1 Material

Four iron powders provided by POMETON with different particle properties were previously examined in a small-scale batch reactor located at Technical University of Darmstadt (TUDA). Among them, Ferelet MT 425/3.0 was selected as the best candidate to be tested in the medium scale fluidized bed reactor at ICB-CSIC (see Figure 1). The first number in the name indicates the maximum permissible particle size and the second number is the apparent density. The total amount of material received from POMETON to conduct the experimental campaigns was 700 kg.

Ferelet MT 425/3.0 description by POMETON

This powder features an irregular morphology with a relatively low oxygen content (0.52%). Its specific surface area supports high oxidation kinetics, making it an ideal candidate for research-oriented or batch-based combustion systems where fast ignition and reactivity are prioritized. However, its lower packing density and moderate flow properties may limit its effectiveness in continuous feed systems.



Figure 1. Ferelet MT material provided by POMETON.

Table 1 compiles the main physical properties and Particle Size Distribution (PSD) data of the Ferelet MT material.

Table 1. Particle size distribution and physical properties of Ferelet MT.

Material	d10 (μm)	d50 (μm)	d90 (μm)	d32 (μm)	Solid density (kg/m^3)	Bulk density (kg/m^3)
Ferelet MT	119.52	181.06	255.65	172.5	7424	3088

2.2 Facility description

The experimental 3 kWth installation consisted of a bubbling fluidized bed (BFB) combustor and different auxiliary systems for gas supply, solid feeding, solid recovering, and gas analysis. Figure 2 shows a schematic diagram of the installation.

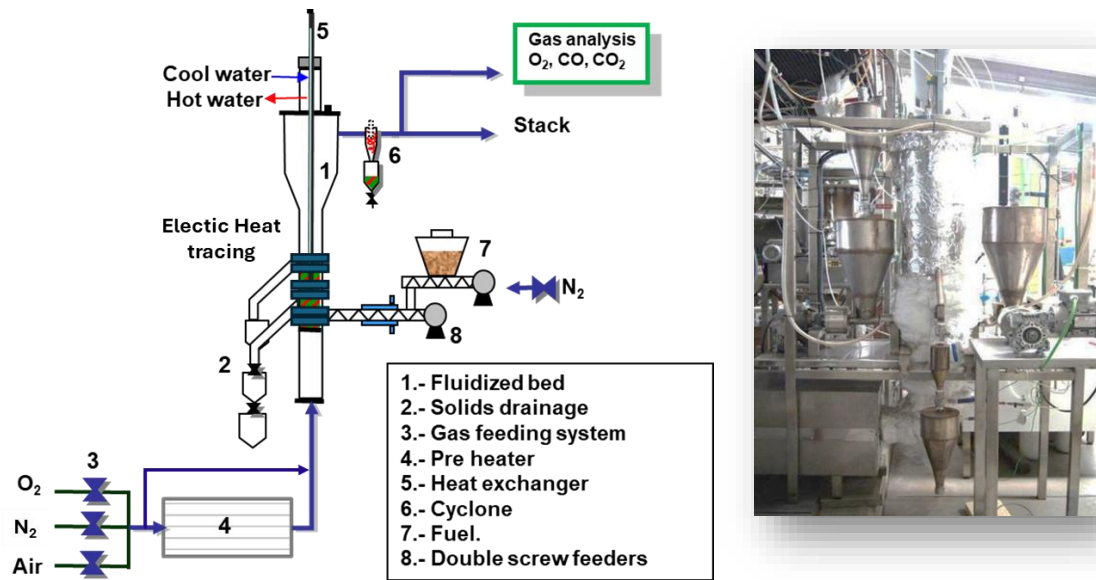


Figure 2. General scheme and photograph of the 3kWth Fluidized Bed Reactor located at ICB-CSIC.

The combustor consisted of a stainless-steel reactor of 9.5 cm i.e. and 62 cm height and a freeboard of 15 cm i.e. and 45 cm height. The operation in the BFB can be performed at two solids bed heights (h_{bed}), 24 and 39 cm. A more detailed information about the different measurements of the reactor is shown in Figure 3.

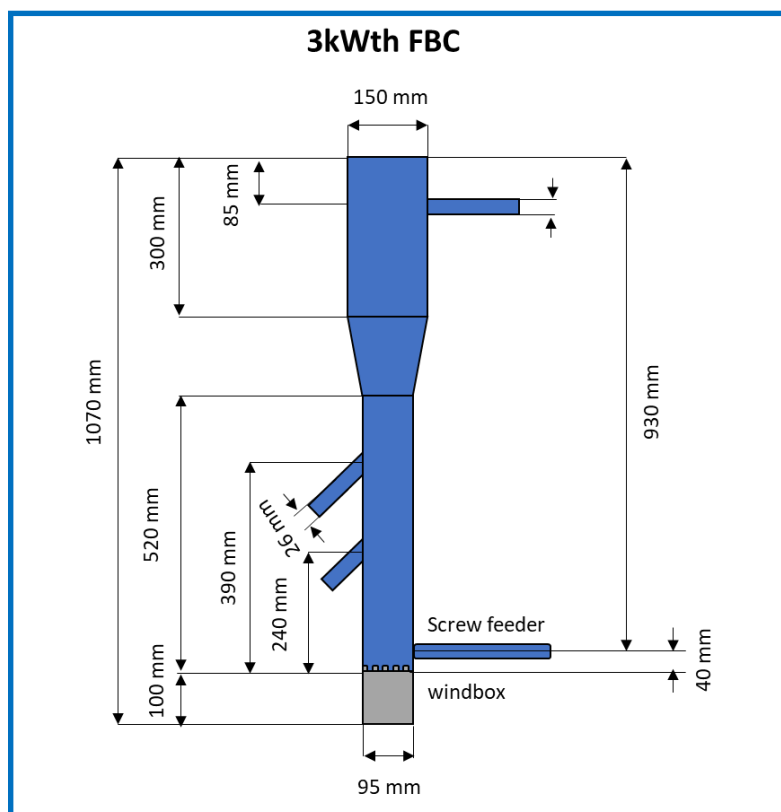


Figure 3. Measurements of the medium-scale FB reactor located at ICB-CSIC.

The reactant gases, air and N_2 , were supplied from bottles by mass-flow controllers and fed into the reactor through a distributor plate. A linear gas velocity of 45 cm/s (calculated at 700 °C) at the inlet of the bed was used during most of the tests. A gas pre-heater allowed introducing hot gas inside the reactor during the start-up of the plant. In addition, an additional heating system was installed, consisting of electrical heat tracing surrounding the reactor. This system helps to achieve the temperature required to initiate material oxidation.

A heat exchanger located inside the bed allowed for the control of temperature during oxidation tests. This heat exchanger could be moved vertically through the reactor, modifying the contact surface inside the bed and extracting the needed heat from the combustor to reach the desired temperature. Additionally, there was also the possibility of bypassing the preheater outlet gas in order to cool down the bed rapidly if necessary.

The solids were fed to the reactor by means of water-cooled screw feeders located just above the distributor plate. To assure a constant fed of the iron material, two screw feeders in series were used. The first one controlled the iron material feeding rate and the second one introduced the solid as quick as possible into the bed to avoid the plugging of the pipe. A N_2 gas flow of 0.6 L/min (STP) was introduced to assist the iron material feeding and prevent backflow of the fluidizing gas towards the feeding system.

The flue gas stream leaving the combustor passed through a high efficiency cyclone to recover the elutriated solids and then was sent to the stack. The gas composition, especially O_2 , at the exit of the combustor was analyzed continuously by a paramagnetic analyzer (Siemens/Oxymat 6). Additionally, NO_x was measured by a mass spectrometer Omnistar Pfeiffer at 750 °C to evaluate the thermal NO_x formation. In this regard, no thermal NO_x formation from N_2 coming from air were detected (< 1 ppm).

The facility was equipped with pressure sensors and thermocouples to know the pressure drop and temperatures along the installation. General process data (temperatures, mass flow of gases, gas composition, etc.) were continuously recorded by a computer.

2.3 Procedure

2.3.1 Bed material preparation

Prior to the fluidized bed combustion experiments with iron as the bed material, a pre-oxidation stage was conducted in the experimental facility. The objective of this procedure was to establish an initial inventory of oxidized iron, thereby enabling better control of heat release during continuous operation, in which reduced iron and air were simultaneously fed into the reactor. Initially, the bed was filled with ~3.5 kg of Ferelet MT (as received).

In a first attempt, the oxidation was carried out using air as the oxidizing gas. It was observed that when the bed temperature reached approximately 800°C, a rapid and uncontrolled temperature increase occurred, reaching values near 940°C. This thermal runaway resulted in severe agglomeration of the bed material, compromising the fluidization quality and preventing the successful completion of the oxidation process. A good temperature control is then particularly important in fluidized beds since high temperatures can promote particle sintering and agglomeration, leading to poor fluidization behavior and operational issues. The condition of bed and the bell-type distributor plate after the experiment is shown in the Figure 4a and 4b, respectively. The plate became completely unusable for subsequent tests. To prevent similar operational issues, the distributor plate was replaced by a flat perforated plate (see Figure 4c) that could be more easily cleaned of the adhered material.



Figure 4a. Bell-type distributor plate with melting material.



Figure 4b. Agglomerates extracted from the bed.



Figure 4c. New flat perforated plate for oxidation tests.

Based on these observations, to avoid an excessive temperature rise during the oxidation process, the oxidizing gas was initially supplied in a diluted form, starting with an oxygen concentration of 5 vol.% and gradually increasing it to 21 vol.%, corresponding to atmospheric air. This approach allowed the oxidation rate of metallic iron to be moderated during the start-up phase, preventing rapid exothermic oxidation and the associated temperature increase within the reactor. By following this approach, the bed temperature was kept below 800°C during the entire oxidation process, thereby preserving the integrity of the fluidized bed. Post-experimental inspection of the bed material showed no evidence of agglomeration. Consequently, the pre-oxidation process was carried out successfully under the selected operating conditions. An example of the oxygen concentration ramp used during the pre-oxidation stage is shown in Figure 5.

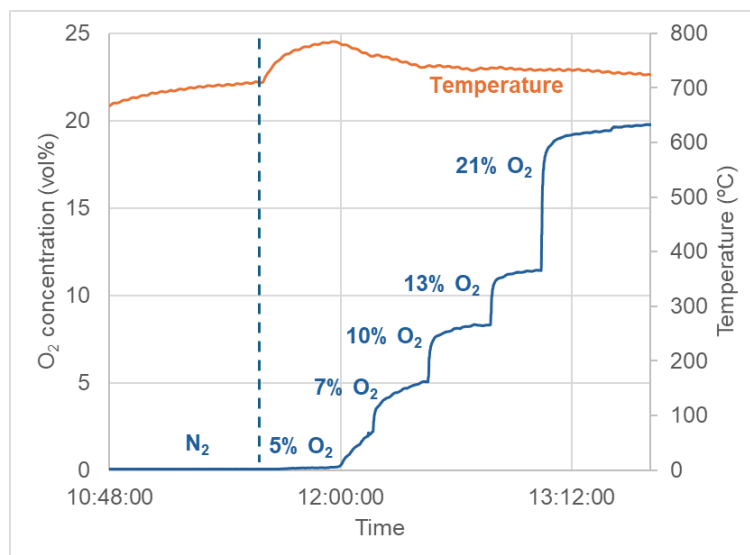


Figure 5. Temperature and oxygen concentration profile during the pre-oxidation stage for bed material preparation.

2.3.2 Continuous feeding tests

Once a partially oxidized bed inventory had been prepared, continuous fluidized bed combustion experiments were started. In these tests, Ferelet MT material was continuously fed into the reactor while air was used as the fluidizing gas.

The start-up procedure began by heating the bed under nitrogen fluidization in order to avoid iron oxidation during this stage. A gas preheater and an electric heater surrounding the reactor were used to increase the bed temperature up to approximately 600 °C, at which iron starts to react significantly with oxygen. At this point, the fluidizing gas was switched from nitrogen to air, and the continuous feeding of iron particles was started using the double-screw feeding system.

The oxygen concentration at the reactor outlet was continuously monitored. The total time for the start-up of the plant was about 2-3 h. Once stable operating conditions were reached, the gas preheater was turned off and the bed temperature was controlled by the heat exchanger and/or the electric heater surrounding the reactor. Steady-state operation conditions were achieved when both the target operating temperature and a constant outlet oxygen concentration were maintained over time. Figure 6 shows the typical evolution of temperature and oxygen concentration during the start-up of the installation until reaching stable operating conditions.

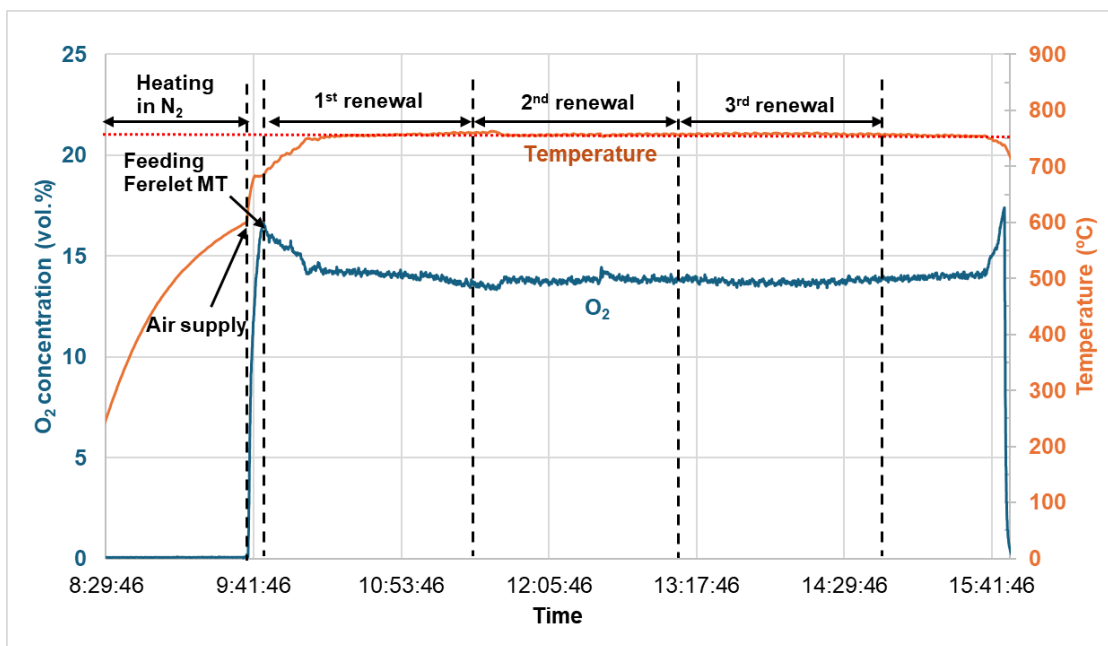


Figure 6. Temperature and oxygen concentration profile of a test conducted at 750 °C with a feeding rate of 2220 g/h and a bed height of 39 cm.

During the continuous experimental tests, several operational challenges had to be addressed. One of the main challenges was temperature control inside the reactor. This issue was successfully managed by using the heat exchanger and by bypassing the gas stream supplied by the preheater, allowing cold gas to enter the reactor whenever additional cooling was required. As a result, the reactor temperature could be maintained within the desired operating range, preventing excessive temperature rise caused by exothermic reaction.

Another important challenge was the continuous feeding of the material using a screw feeder. The screw frequently tended to become stuck during operation. Once it had been stopped, it could not be restarted again. This behavior was attributed to the mechanical properties of the material being fed, particularly due to its high hardness compared to other materials commonly tested in the pilot plant, such as biomass or coal. Because of this higher hardness, particles could become trapped between the screw flight and the wall of the conveying tube, preventing the screw from rotating. Under these conditions, the torque provided by the motor was not sufficient to crush the trapped particles or overcome the resistance generated by the blockage.

To overcome this issue, the existing screw feeder was redesigned by modifying its outer diameter, flight depth, and pitch distance between flights. These modifications enabled a stable and reliable continuous feeding operation throughout the experiments.

2.3.3 Steady state conditions

An important feature of the tests carried out in continuous units is the certainty that the results are obtained under steady state conditions, which must consider the mean residence time of solids. In the present facility, the solids residence time is controlled by both the bed inventory and the solids feeding rate, which is directly associated with the Ferelet MT withdrawal rate through the drainage point by overflow ($\dot{m}_{FeMT,out}$). The position of this point determines the operating bed height and, consequently, the bed inventory (m_{bed}). Two bed heights were investigated in this work, namely 24 cm and 39 cm, corresponding to bed inventories of approximately 2.5 kg and 4 kg, respectively. This configuration provides considerable flexibility for studying a wide range of solids residence times, between 1 and 3 hours.

The mean solids residence time in the bed was estimated using equation (1):

$$\tau \text{ (h)} = \frac{m_{bed}}{\dot{m}_{FeMT,out}} \quad (1)$$

To be sure that steady state conditions had been reached, it was considered that the weight of solids extracted from the bed and collected in the drainage deposits should be at least 2–2.5 times the weight of solids existing in the bed (m_{bed}). Accordingly, once stable operating conditions had been established, a steady outlet oxygen concentration was typically achieved after 5–7 h of operation for each experimental test.

2.3.4 Mass Balance

The oxidation degree of the particles was determined by equation (2) from oxygen mass balance.

$$X_{FeMT} = \frac{n_{FeMT,reacted}}{n_{FeMT,in}} = \frac{(n_{O2,in} - n_{O2,out})}{\left(\frac{\dot{m}_{FeMT,in}}{M_{M,Fe}}\right)} \quad (2)$$

where $n_{FeMT,reacted}$ is the moles of Ferelet MT reacted, $n_{FeMT,in}$ is the moles of Ferelet MT fed to the reactor, $n_{FeMT,in}$ and $n_{O2,out}$ are the moles of oxygen at the inlet and outlet of the reactor, respectively, and $M_{M,Fe}$ is the mass molar of Ferelet MT assuming that is pure iron.

Samples collected under steady-state conditions were analyzed by thermogravimetric analysis (TGA) to verify the particle oxidation degree values obtained from the oxygen mass balance. For this purpose, the samples were subjected to a reduction step at 950 °C using a 15% of H₂ (N₂ to balance) as reducing gas. Figure 7 shows a typical thermogram obtained during this procedure. The conversion of Ferelet MT ($X_{ox,TGA}$) was calculated using equations (3)-(4)

$$X_{ox,TGA} = \Delta m / \Delta m_{max} \quad (3)$$

$$\Delta m_{max} = m_{ox} - m_{red} \quad (4)$$

Where m_{ox} is the mass of the material totally oxidized to Fe_2O_3 and m_{red} is the mass of the material totally reduced to Fe.

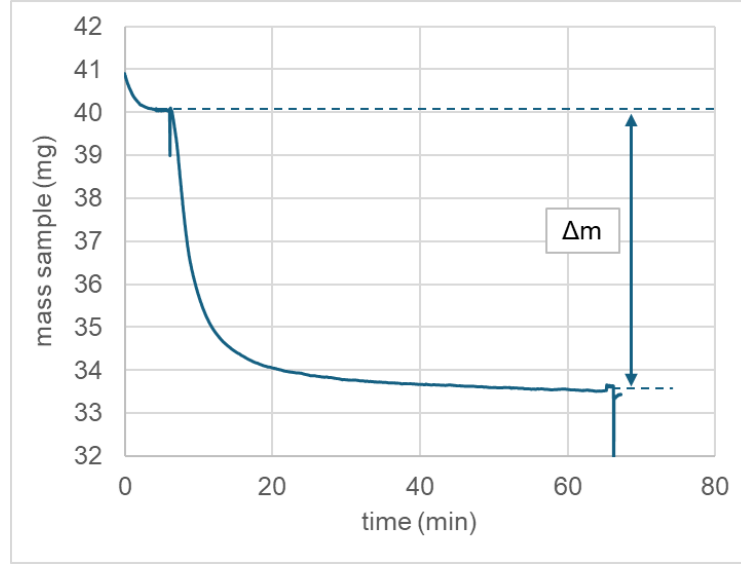


Figure 7. Example of a reduction test performed in TGA to determine the oxidation degree of the bed particles. $T = 950\text{ }^{\circ}\text{C}$; 15 vol.% H_2 (N_2 to balance).

Additionally, fines generation and changes in the particle size distribution of the material during operation were studied. These results will be further discussed in the subsequent sections 3.5.2 and 3.5.3, respectively.

3 Results

To determine the optimal operating window for the oxidation stage, several experimental campaigns were carried out to evaluate the effect of the main operating variables on the oxidation degree of the iron material (solids mean residence time, temperature, gas velocity, air ratio).

The range in the feeding rate is constrained by the operational limits of the medium-scale BFB combustor. On one hand, the thermal capacity of the plant (3 kWth) sets an upper limit on the amount of material that can be processed (around 2500 g/h) and on the other hand, the performance of the screw-feeding system defines the lower limit, as there is a minimum feeding rate that can be maintained in a stable and reliable manner (around 1100 g/h).

Due to the characteristics of the material, particularly its high density and broad particle size distribution, a superficial gas velocity of 45 cm/s was selected as the reference operating condition. This fluidization velocity was chosen to ensure adequate fluidization of the larger particles while minimizing the entrainment and elutriation of the finer fractions from the reactor. Consequently, stable fluidization conditions could be maintained across the entire particle size range, reducing solids losses and ensuring a representative evaluation of the oxidation performance of the material. In addition, experiments were also conducted at superficial gas velocities of 35 and 55 cm/s in order to evaluate the influence of the air ratio (λ) on the oxidation performance of the material. This air ratio was calculated as the ratio between the total oxygen supplied to the reactor and the stoichiometric oxygen required for the complete oxidation of iron to iron (II) oxide, as defined in Equation (5).

$$\lambda = \frac{O_{2,supplied}}{O_{2,stoich}} \quad (5)$$

In addition, the experiments were carried out within the temperature range of 700-800 °C. This range was selected to ensure that the material exhibited high enough reactivity while reducing the risk of agglomeration issues.

Table 2. presents a summary of the operating conditions and variables evaluated during the experimental campaign as well as the O₂ in the flue gas and oxidation degree of the material obtained by mass balance at both bed heights, 39 and 24 cm.

Table 2. Summary of testing conducted in the medium scale FB combustor at two bed heights (39 and 24 cm).

$h_{bed} = 39$ cm									
T (°C)	Solids feeding rate $\dot{m}_{FeMT,in}$ (g/h)	Gas velocity (cm/s) *	Solids inventory, m_{bed} (kg)	Solids mean residence time, τ (h)	Air ratio, λ (-) *	O _{2,out} (vol.%)	X_{FeMT} (-)	$X_{ox,TGA}$ (-)	Error (%)
800	1149	45	3663	2.46	1.96	14.3	0.69	0.763	10.5
	1573	45	3653	1.78	1.43	11.8	0.70	0.723	3.3
	1843	45	3740	1.60	1.22	11.5	0.623	0.615	1.3
	2432	45	3793	1.25	0.92	9.2	0.567	0.594	4.8
	1885	35	3723	1.55	0.91	7.7	0.631	0.626	0.8
	1870	55	3760	1.60	1.47	13.5	0.597	0.623	4.3
750	1142	45	3096	2.84	1.97	16.6	0.475	0.48	1.0
	1438	45	3945	2.30	1.53	15.8	0.445	0.464	4.3
	2000	45	4030	1.72	1.12	14.7	0.385	0.42	9.1
	2220	45	4023	1.55	1.01	13.9	0.39	0.377	3.3
700	1220	45	4164	3.03	1.84	18	0.30	0.30	0
	1450	45	4172	2.54	1.55	17.8	0.295	0.327	10.8
	1760	45	4239	2.17	1.28	17.4	0.257	0.282	9.7
	2050	45	4248	1.87	1.1	16.8	0.252	0.265	5.2
	1826	335	3938	1.93	0.96	15.7	0.275	0.293	6.5
	1625	55	3938	2.12	1.69	17.8	0.30	0.30	0
$h_{bed} = 24$ cm									
T (°C)	Solids feeding rate $\dot{m}_{FeMT,in}$ (g/h)	Gas velocity (cm/s) *	Solids inventory, m_{bed} (kg)	Solids mean residence time, τ (h)	Air ratio, λ (-) *	O _{2,out} (vol.%)	X_{FeMT} (-)	$X_{ox,TGA}$ (-)	Error (%)
800	1300	45	2361	1.48	1.73	15.6	0.51	0.526	3.1
	1500	45	2376	1.30	1.50	14.7	0.51	0.511	0.2
	1950	45	2239	0.95	1.15	13.1	0.49	0.466	4.9
	2550	45	2291	0.76	0.88	12	0.422	0.452	7.3
750	1825	45	2547	1.23	1.23	16.3	0.315	0.325	3.2
700	1825	45	2657	1.33	1.23	17.8	0.215	0.241	12.1

* Determined at 700°C

3.1 Effect of solids feeding rate

The effect of varying the solids feeding rate under a constant fluidization velocity of 45 cm/s was first investigated using a bed height of 39 cm. Figure 8 illustrates the influence of the solids feeding rate in the oxidation degree of the material, which decreases as the amount of solids fed into the reactor increases.

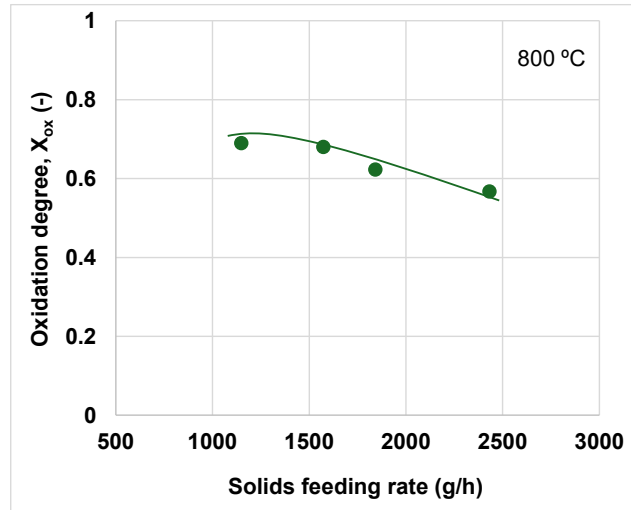


Figure 8. Effect of the solids feeding rate on the oxidation degree of the bed particles at 800 °C and a gas velocity of 45 cm/s with a $h_{bed} = 39$ cm.

Since the total gas flow rate and the inlet oxygen concentration were kept constant throughout all experiments, i.e. air was always used as the fluidizing gas, changes in the solids feeding rate inherently led to different operating conditions in terms of excess or deficiency of oxygen (λ) and mean solids residence time.

Figures 9a and 9b depict the oxidation conversion of the material as a function of the air ratio and mean solids residence time, respectively at 800 °C. As can be observed, the oxidation conversion exhibits a similar dependency on both variables. Higher air ratio values and longer mean solids residence times lead to increased oxidation conversions. This behavior can be attributed to the greater oxygen availability and/or the extended gas–solid contact time, which promote the progress of the oxidation reaction and enhance the overall conversion of the material.

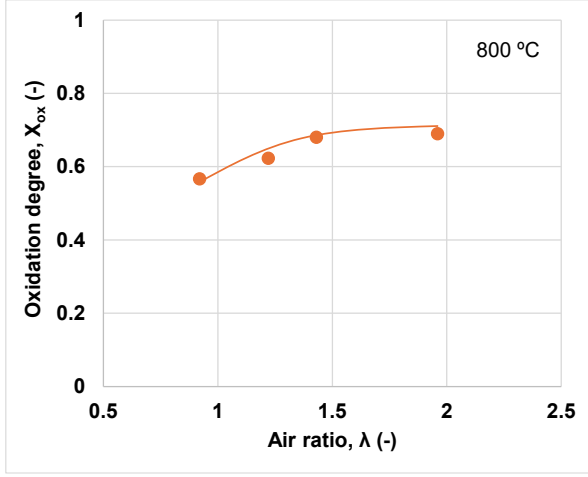


Figure 9a. Oxidation degree of the bed particles as a function of the air ratio at 800 °C and a gas velocity of 45 cm/s with a $h_{bed} = 39$ cm.

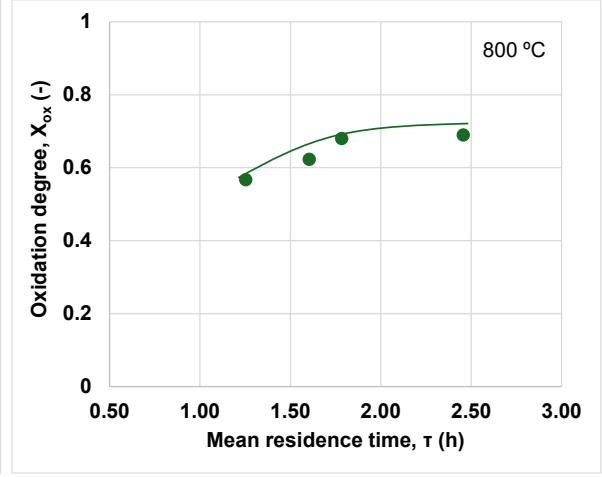


Figure 9b. Oxidation degree of the bed particles as a function of the mean solids residence time at 800 °C and a gas velocity of 45 cm/s with a $h_{bed} = 39$ cm.

3.2 Effect of the air ratio, λ

As both the air ratio and mean solids residence time vary simultaneously in the experiments presented above, their individual effects on the oxidation conversion cannot be directly distinguished. Therefore, it is necessary to assess the relative importance of each parameter in determining the oxidation degree of the material. Identifying the dominant variable governing the oxidation conversion is crucial for establishing the primary factor controlling the oxidation performance of the material under the studied operating conditions.

In order to decouple the influence of the air ratio and solids residence time, the effect of the air ratio was evaluated independently by varying the superficial gas velocity while keeping the solids feeding rate constant at approximately 1900 g/h. Three gas velocities, namely 35, 45, and 55 cm/s, were tested. As illustrated in Figure 10, only minor differences in oxidation conversion were observed despite the changes in the air ratio performed at two different temperatures, 700 and 800 °C. This finding indicates that, within the range investigated, the oxidation degree is not significantly limited by the oxygen availability. Consequently, it can be concluded that mean solids residence time has more effect on controlling the oxidation degree of the Ferelet MT material. Therefore, for a given temperature, extending the residence time of the particles within the reactor is more effective than increasing the air ratio to achieve a higher oxidation conversion.

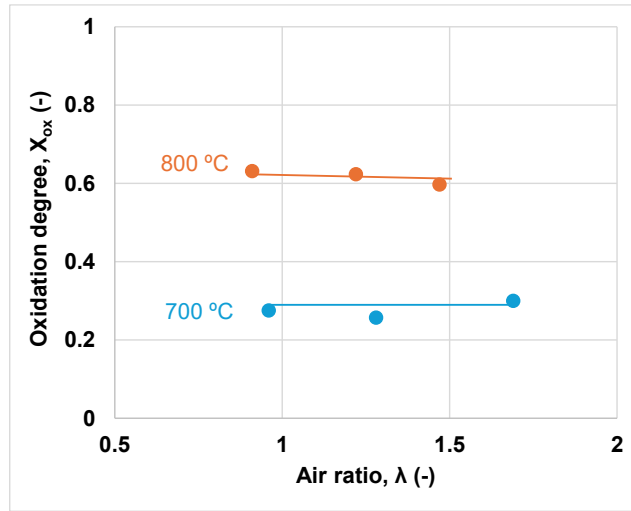


Figure 10. Effect of the air ratio on the oxidation degree of the bed particles at 700 and 800 °C and different gas velocities (35-45-55 cm/s) with a $h_{bed} = 39$ cm.

3.3 Effect of the operating temperature

The influence of temperature on the oxidation performance of the Ferelet MT material was also studied by conducting a series of experiments between 700 and 800 °C with a bed height of 39 cm. These tests were performed for all the solids feeding rates considered in this study in order to evaluate the combined effect of temperature and mean solids residence time on the oxidation conversion. The results are presented in Figure 11. As expected, an increase in temperature enhanced the oxidation process, resulting in higher conversion values at a given residence time. This behavior is attributed to the faster oxidation kinetics at elevated temperatures, which increase the reaction rate and allow a greater extent of conversion to be achieved within the reactor.

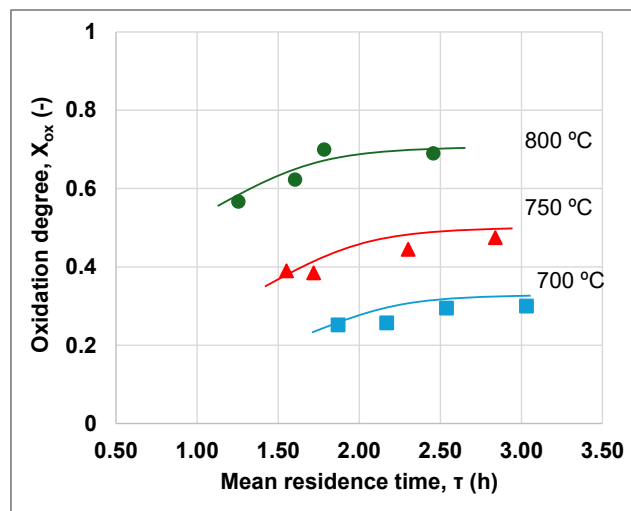


Figure 11. Effect of the temperature on the oxidation degree of the bed particles for different solids mean residence times operating with a $h_{\text{bed}} = 39$ cm.

Upon completion of the experimental campaign at every temperature, the reactor and bed inventory were inspected to assess the physical condition of the material. After operation at 800 °C, significant agglomerate formation was observed, with the presence of large, consolidated particle clusters within the bed; see Figure 12. These observations indicate that operating at 800 °C promotes agglomeration phenomena, likely as a result of particle sintering at elevated temperatures. Since agglomeration can compromise fluidization quality and eventually lead to operational problems, temperatures close to 800 °C are not recommended for the oxidation of Ferelet MT despite the enhanced reactivity achieved. Consequently, the oxidation of the material should preferably be carried out at temperatures below 800 °C to ensure stable operation and preserve the integrity of the material.



Figure 12. Image of agglomerates extracted from the bed after inspection when operating at 800 °C and a bed height of 39 cm.

Post-experimental inspection of the bed inventory after the campaigns carried out at 700 and 750 °C revealed the formation of small agglomerates in both cases; see Figure 13. Nevertheless, the degree of agglomeration was limited and did not compromise reactor operability or fluidization quality. The absence of significant operational issues indicates that the material can be operated within this temperature range. Therefore, although some degree of particle interaction occurs, temperatures between 700 and 750 °C provide a suitable balance between

oxidation performance and resistance to agglomeration. It is worth noting that the agglomerates were mainly located near the edges of the distributor; see Figure 14. These regions exhibited poorer fluidization conditions due to the limited number of distributor holes, which resulted in inadequate gas distribution and reduced solid movement. The presence of these poorly fluidized zones likely favored particle accumulation and prolonged particle–particle contact, enhancing the formation of local agglomerates.



Figure 13a. Image of agglomerates extracted from the bed after inspection when operating at 750 °C and a bed height of 39 cm.



Figure 13b. Image of agglomerates extracted from the bed after inspection when operating at 700 °C and a bed height of 39 cm

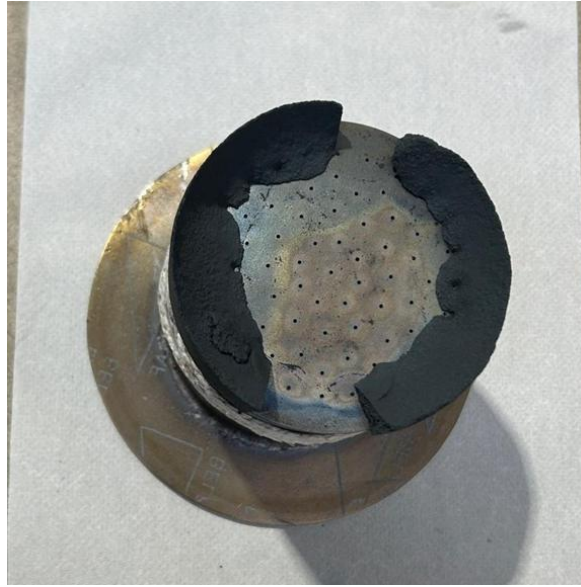


Figure 14. Image of agglomerates found around the distributor plate after inspection when operating at 750 °C and a bed height of 39 cm.

3.4 Comparison of results at two bed heights

The mean residence time of solids is affected by the solids feeding rate and the solids inventory in the reactor. In order to evaluate the effect of the solids inventory, tests at two different bed heights, namely 39 and 24 cm, were performed. In addition, the solids feeding rate was varied in both cases. It was observed that for the lower bed height, lower oxidation conversions were obtained; see Figure 15a. This behavior is because of the different mean residence times in every condition; see Figure 15b. Comparing the results as a function of the solids mean residence time, it was observed that data obtained at 24 cm corresponds properly with the trend shown at 39 cm.

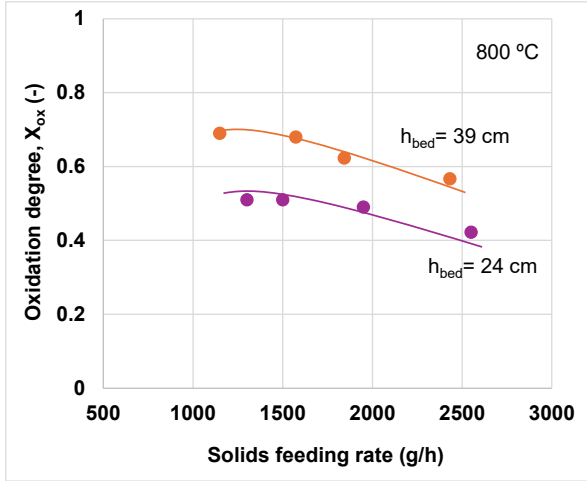


Figure 15a. Comparison of the effect of the solids feeding rate on the oxidation degree of the bed particles at 800 °C for both bed heights, 24 and 39 cm, and operating with a gas velocity of 45 cm/s.

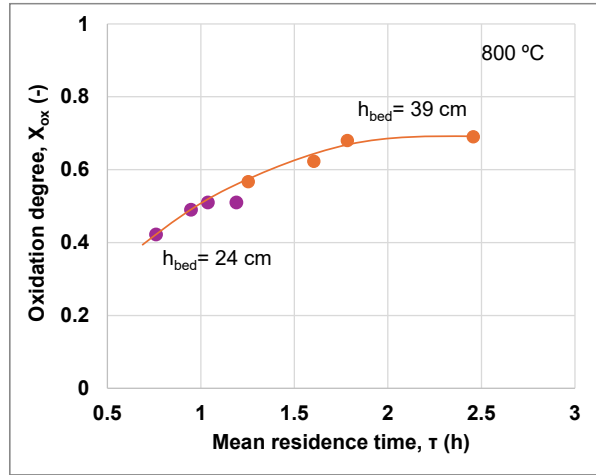


Figure 15b. Effect of the solids mean residence time on the oxidation degree of the bed particles at 800 °C operating at both bed heights, 24 and 39 cm, and a gas velocity of 45 cm/s.

The effect of temperature was also evaluated at the lower bed height. As can be seen in Figure 16, lower oxidation conversions compared to those achieved at the higher bed height, i.e. 39 cm, were obtained for the entire temperature range, mainly because of the lower values of solids mean residence times too. It was noted that the differences in oxidation conversion for both bed heights at a given temperature become more pronounced as the reaction temperature increases. Likewise, the results showed that increasing the residence time at the low temperature was insufficient to reach oxidation conversion values comparable to those achieved at higher temperatures, even when significantly shorter residence times were used. These results suggest that temperature has a more pronounced effect on oxidation conversion than residence time within the studied operating range, confirming the strong influence of temperature on the oxidation kinetics of the material.

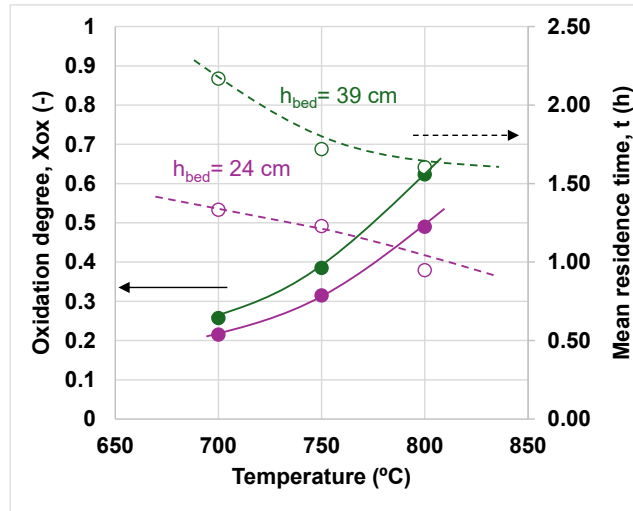


Figure 16. Comparison of the effect of the temperature and solids residence time on the oxidation degree of the bed particles for both bed heights, 24 and 39 cm, and a gas velocity of 45 cm/s.

3.5 Ferelet MT characterization

3.5.1 Validation of solids conversion values with TGA tests

During the experimental campaign, bed material samples were collected under steady-state operating conditions at different reaction temperatures and residence times. The oxidation degree of the samples was subsequently determined by thermogravimetric analysis (TGA).

As can be seen in Figure 17, good agreement was observed between the oxidation degrees measured by TGA and those calculated from the mass balances described in Section 2. This consistency confirms the validity of the mass balance methodology and provides independent verification of the experimental results. In most cases, the deviation between the theoretical oxidation degree and the value obtained by TGA was lower than 10%, demonstrating the high reliability of the oxidation conversion calculations.

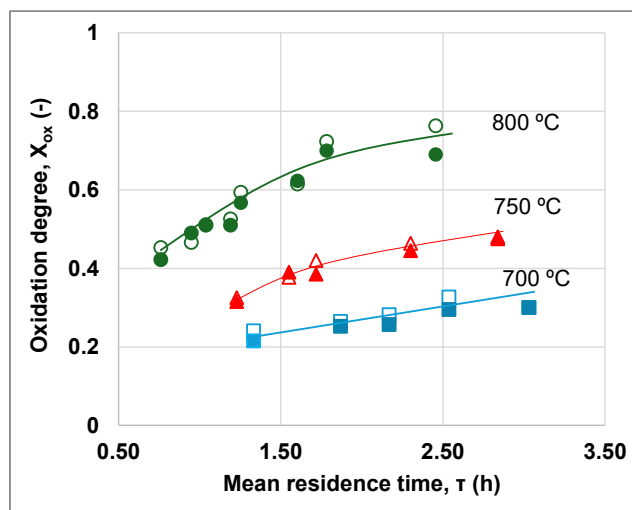


Figure 17. Comparison of the oxidation degree values obtained by oxygen mass balance and TGA at different temperatures and solids residence times. Bed heights = 24 and 39 cm, and gas velocity = 45 cm/s. Empty Symbol: TGA values; Filled Symbol: Mass balance values.

3.5.2 Attrition rate and fines generation

During the experimental campaigns at different temperatures, the generation of fines from bed material and the elutriation rate was investigated. The elutriated rate is calculated as a percentage of grams of fines collected in the cyclone per g of material fed to the reactor during a determined time. The values obtained with a bed height of 39 cm are summarized in Table 3. It was observed that attrition increases by increasing the operating temperature and also by decreasing the feeding rate since it is associated with working at higher oxidation degree and mean residence time. In this sense, it is remarkable the high fraction of fines generated at 800 °C and with the lower solids feeding rate tested, which also improved the solids conversion.

Table 3. Amount of fines (in %wt of the solids fed) collected from the cyclone during the tests performed at different temperatures and solids feeding rates with a bed height of 39 cm and gas velocity of 45 cm/s.

Temperature (°C)/ Feeding rate (g/h)	800	750	700
	%wt fines		
1200	8.5	3.3	2.6
1500	3.0	2.4	2.1
1900	3.3	4.5	1.7
2400	4.0	1.5	1.5

3.5.3 Particle Size Distribution

After every test, bed samples were extracted and characterized to determine their particle size distribution. The results showed a clear decrease in average particle size with increasing feeding

rate operating at a given temperature. This behavior can be attributed to the lower oxidation conversion achieved at higher feeding rates, which limits the extent of particle growth associated with the oxidation process. Consequently, particles experience a lower degree of growth, resulting in a smaller average particle size in the bed material. Figure 18 shows the different PSD obtained when operating at 800 °C.

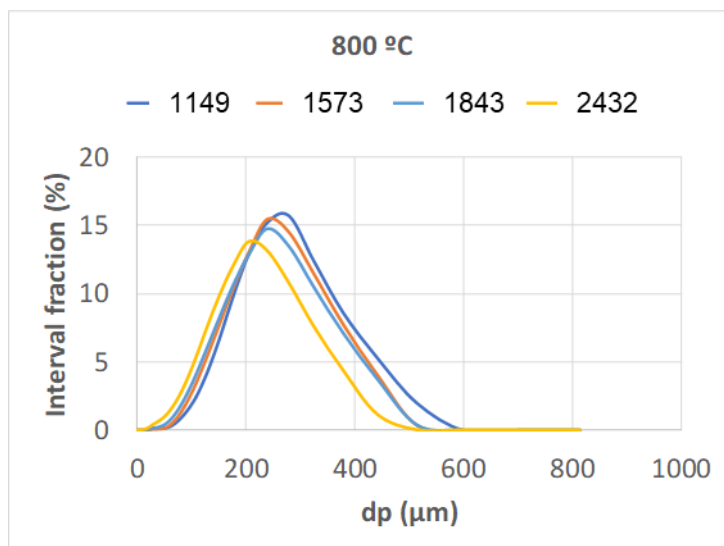


Figure 18. Particle Size Distribution of the samples extracted from the bed at 800 °C for different solids feeding rates (g/h).

Figure 19a shows the mean particle size profile obtained for all bed samples collected from the reactor at different temperatures and solids feeding rates, while maintaining a constant gas velocity of 45 cm/s. The solid blue line represents the expected particle size growth as a function of oxidation conversion of the particle, calculated considering the difference in the molar volume between the iron and iron oxide and a slight increase in the particle porosity during oxidation.

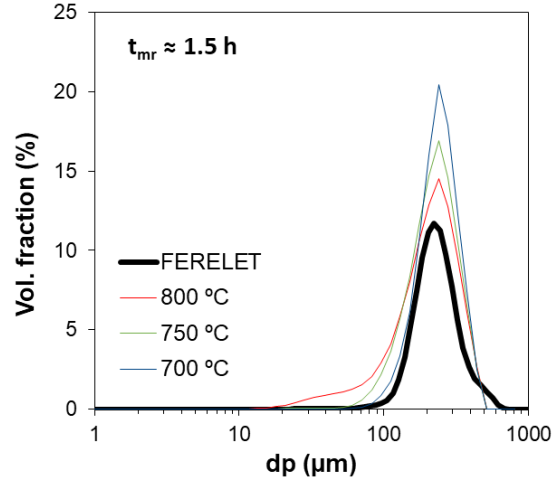
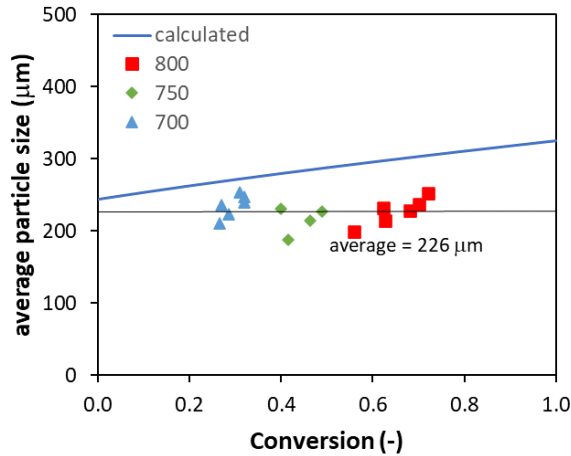


Figure 19a. Average particle size as a function of the conversion degree of the material. Solid line: theoretical evolution of particle size. Symbols: experimental values obtained at different temperatures and solids feeding rates. Bed height: 39 cm and gas velocity = 45 cm/s.

Figure 19b. Comparison of Particle Size Distribution of samples with similar mean residence time (≈ 1.5 hours) at different temperatures and the fresh material.

As can be observed, the average particle size would be expected to increase by increasing the conversion due to the particle growth (blue solid line). However, the mean particle size remains nearly constant at approximately 226 μm . This behavior can be mainly attributed to an abrasion mechanism on the particle surface, which generates fines. Some fine particles were elutriated and recovered in the cyclone, while another fraction remains still in the bed. As shown in Figure 19b, the amount of fines being accumulated in the bed increases with temperature and therefore, although the particle size tends to increase because of the higher oxidation conversion achieved at higher temperatures, this growth is offset by the simultaneous generation of fines. As a result, the average particle size remains practically unchanged.

4 Lesson learnt and conclusions

During the oxidation tests of Ferelet MT material in the middle-scale 3 kWth unit, maintaining temperature control at 800 °C was found to be particularly challenging due to the significant increase in oxidation kinetics at this temperature. Once the oxidation reaction was initiated, a rapid temperature rise was observed, with temperatures potentially exceeding 900 °C. Such temperature increases were identified as a critical factor promoting particle agglomeration and affecting the stability of the process.

The results demonstrate that adequate gas distribution through the distributor plate is a key requirement for maintaining stable fluidization and preventing particle agglomeration. Poor gas distribution was observed to induce agglomeration even at relatively low temperatures (750 °C). Moreover, complete interruption of the fluidizing gas flow, such as that caused by an electrical failure, led to rapid agglomeration of the bed material. These observations underline the importance of ensuring robust fluidization conditions and implementing appropriate process safeguards to avoid bed defluidization and operational failure.

Based on the results presented herein, an appropriate balance must be established between operating temperature and material oxidation capacity. While increasing the temperature enhances oxidation rates, it also significantly increases the risk of agglomeration and operational instability. Considering both oxidation performance and bed stability, an optimal operating temperature range of 700–750 °C is recommended for this material. In addition to mitigating agglomeration issues, operation within this range reduces attrition of the material, contributing to improved process efficiency and extended material lifetime.

Finally, it should be noted that, among the operating variables investigated for the oxidation process of this material, temperature was identified as the most influential parameter affecting the particle oxidation degree, followed by mean residence time and the air ratio. Therefore, the relative importance of the variables can be ranked as follows, from highest to lowest influence: temperature > mean residence time > air ratio.