

Grant Agreement Number:
641185

Action acronym:
CEMCAP

Action full title:
CO₂ capture from cement production

Type of action:
H2020-LCE-2014-2015/H2020-LCE-2014-1

Starting date of the action: 2015-05-01
Duration: 42 months

D8.3

Assessment of calciner test results Revision 1

Due delivery date: 2017-12-31
Actual delivery date: 2018-3-31

Organization name of lead participant for this deliverable:
C.T.G. SPA

Project co-funded by the European Commission within Horizon2020		
Dissemination Level		
PU	Public	X
CO	Confidential , only for members of the consortium (including the Commission Services)	

Deliverable number:	D8.3
Deliverable title:	Assessment of calciner test results
Work package:	WP 8 Calciner Technology for Oxyfuel Process
Lead participant:	CTG

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Keywords
Calciner, Oxyfuel, Calcination degree, Calcination temperature

Abstract
<p>Between the different calciner configurations, described in D8.1, the in-line calciner is the best option for the operation in oxyfuel mode. A few considerations are reported to confirm that the calciner rig in Stuttgart University represents quite closely the industrial situation both in air calcination mode and in oxyfuel mode; the same for the difficult task to measure real values of temperature, inevitably biased by radiant heat, because it is possible to assume that similar deviations are included in the industrial measured values; the difference between the real temperature value and the measured one should not be higher than 40 °C. Consequently it is possible to consider the results obtained by the tests done in work package WP8 in different conditions as meaningful and useful to supply quantitative information to be used in the mathematical model developed in WP6. Effects on calcination degree consequent to temperature, residence time, CO₂ concentration, raw meal fineness, water vapor have been evaluated. The main finding is that decarbonation takes place in an oxyfuel mode at temperatures that are higher than those registered for air calcination: the difference is in the order of 50 °C, and the temperature absolute value of calcination in oxyfuel mode is evaluated to be around 920 °C. This value is critical from the technological point of view and may be a possible cause of anomalous coatings. This aspect can't be analyzed with the available equipment and will be matter of studies during future full scale tests. The other parameters have also influence on the calcination process but all of them need the support of temperature to reach an acceptable degree of calcination.</p>

Please cite this report as: [Cinti Giovanni], [2017]. [Assessment of calciner test results(D8.3)]. Refer to the [CEMCAP community in Zenodo.org](https://zenodo.org/communities/CEMCAP) for citation with DOI.

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1 INTRODUCTION

Calciner technology, introduced for the first time in 1973, has been developed since then trying to increase the kiln line performance in terms of energy consumption, pollutant emission reduction and alternative fuels combustion. As widely treated in D 8.1, today many different kinds of calciners are available, having in common the basic scope of performing the dissociation of the limestone in calcium oxide and carbon dioxide.

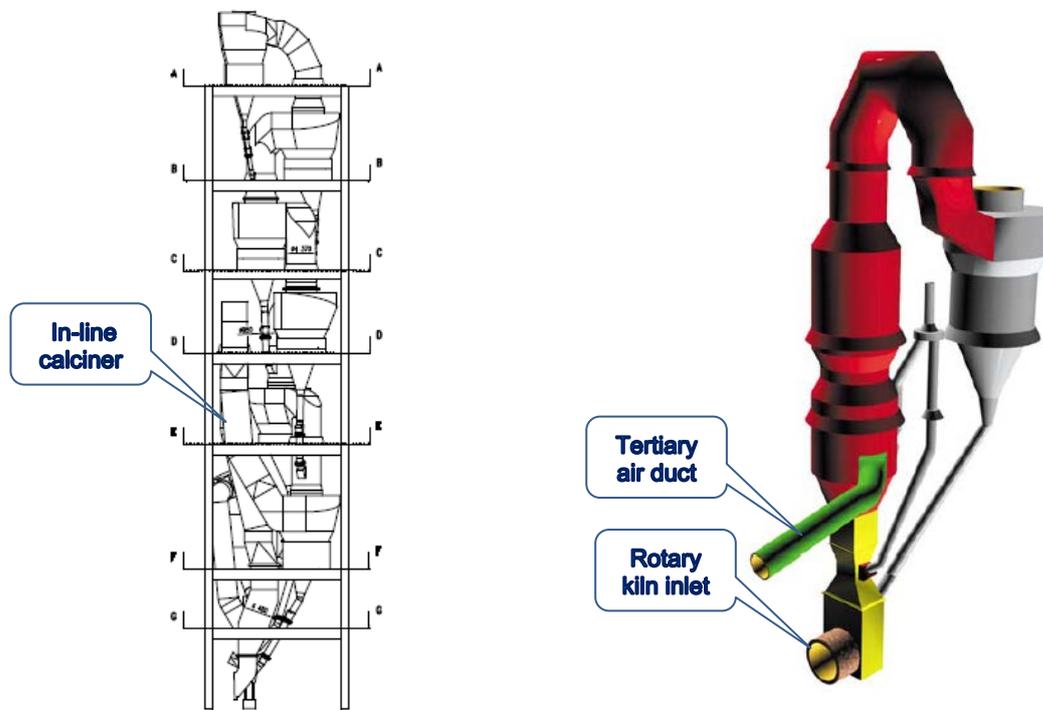


Fig. 1.1 : Five stage preheater kiln with in-line calciner

The most common calciner configuration is the so-called in-line calciner; in this case the calciner is located at the end of the rotary kiln at the base of the preheater tower. In this configuration the gas coming from the rotary kiln enters at the bottom of the calciner through a restriction where the gas is accelerated up to 30-35 m/s to take in suspension the raw meal coming down from the fourth preheater stage. Over the raw meal injection point, the tertiary air duct provides the combustion air to the burner(s) located at the same level. Part of the raw meal can also be introduced through another injection point to control the temperature in the burning zone.

Many possible different designs are adopted for the fuel burners: in some cases they are just injectors of pulverized fuel, only associated with the necessary amount of transport air, but there are even more complex solutions with primary air split in axial and radial. In any case the combustion takes place without the formation of a flame because the heat which is developed is immediately absorbed by the limestone for the dissociation reaction. The combustion takes place in a diffusive way and is distributed and completed all along the total volume of the calciner. The volume of the calciner itself is sized in such a way that the specific fuel which is adopted can be burnt during the foreseen residence time. Considering a plug flow the residence time goes from 3 s, when only finely ground coal or petcoke are used, up to 6-7 s, which are necessary to guarantee the combustion of alternative solid fuels like shredded tires or solid recovered fuel.

(SRF). Due to the fact that inside the calciner 12-15 m/s of gas speed are necessary to guarantee the raw meal dispersion and transport with the gas, the total length of the calciner can be immediately fixed between 40 and 100 m.

Whatever the design is, the expected degree of calcination of the raw meal entering the rotary kiln is around 90-95%. In a conventional calciner, using tertiary air for the combustion of fuel, the controlling factor of calcination degree is the average temperature inside the calciner, which is depends on many different factors. In case of an oxyfuel combustion an additional important factor is the CO₂ concentration in the calciner atmosphere, as high as 80%. This aspect in particular has been the subject of the investigations done by the University of Stuttgart with the oxyfuel suspension calciner rig.

The test facility is an electrically heated entrained flow reactor, 2.5 m long with an internal diameter of 0.2 m, where preheated limestone finely ground or raw meal is dropped inside from the top, by means of a dosing screw; hot gases are also injected at the top, carrying with them the material at the same speed down to the outlet, so that by changing the total flow rate it is possible to change the residence time inside the reactor. 58 kW of electrical power can provide more than the energy required to reach the calcination temperature and to supply the heat for calcination of limestone.

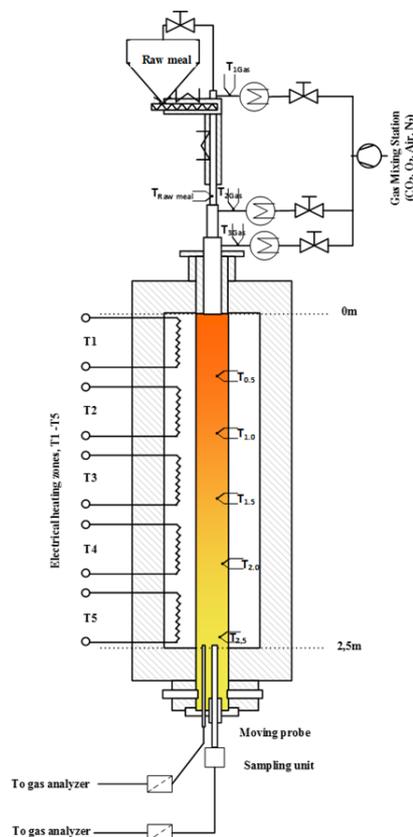


Fig. 1.2 Modified facility suitable for entrained calcination tests

2 ASSESSMENT AND COMPILATION OF WP8 RESULTS FOR FURTHER USE IN WP6 OXYFUEL SIMULATIONS

Theoretical assumption has been initially used as a first approach to operate the model for the oxyfuel simulation developed in WP6. To increase its accuracy it is of course necessary to introduce in the model a number of parameters deduced from specific tests performed in oxyfuel condition regarding the combustion process, the calcination reaction and the clinker cooling conditions.

As far as the calcination reaction is concerned, an overview of the existing calciner technology has been introduced in D8.1. All the main types have been considered, starting from extended riser duct calciners (reported also as “small calciners” because the calcination degree doesn’t exceed the 20%) up to the in line calciners able to control NO_x formation and to burn alternative fuels. These calciners are characterized by a long retention time, between 6 and 8 s, and by the possibility of introducing the raw meal, coming from the upper stage, in a couple of points located at different levels of the calciner; this possibility is mainly connected with the scope of reducing as much as possible the formation of NO_x in the calciner. Even if this problem is probably less important in a calciner operating in oxyfuel mode (very low amount of nitrogen in the combustion atmosphere) it is always important to have the possibility of splitting the raw meal between two possible levels, because it allows also a better control of the temperature level inside the calciner. The calciner of this type is definitely to be considered as the best technology also for future kilns operating in oxyfuel mode; the model adopted this configuration which is therefore confirmed.

Tests to simulate the calciner oxyfuel mode have been already reported by literature:

- Some of them have been done in connection with the calcium looping process for the capture of CO_2 from power plants [1]. These tests are very interesting but are not reproducing the real conditions that we can have in a calciner fitted to a cement kiln. The tests are operated with pure limestone (and not with raw meal, a mixture containing also oxides of Si, Al and Fe) and the granulometry of limestone is much finer than the granulometry in an ordinary raw meal. Nevertheless it has to be underlined that these tests put in evidence a sensible increase of the dissociation temperature of limestone, which in normal operation is around 840-860 °C, while in CO_2 rich atmosphere goes up to 920-950 °C.
- Tests made on a lab scale by ECRA are showing the same behavior [2]; an even higher temperature shift, up to 80 °C, is found with the additional information that not all sources of limestone are effected in the same way by the CO_2 rich atmosphere: in particular Marl, a classical constituent of raw meal naturally mixed with silica oxides, shows a reduced temperature shift than pure limestone.
- Also Cinar, on behalf of ECRA, has studied this phenomena by means of a CFD simulation [3], finding a lower temperature increase of limestone dissociation temperature (about 15 °C) with a consequent slightly reduced degree of decarbonation, from 93% in air mode to 91% in oxyfuel mode, all other parameters unchanged. Along with these modifications other effects have been shown by the CFD simulation: the burning velocity of fuel is higher in the oxyfuel mode, due to higher radiative heat transfer component the heat transfer coefficient is also higher, as well as the residence time, due to the lower decarbonation degree (lower flow rate).
- An oxyfuel test on a pilot plant, jointly made by Air Liquide, FLSmidth and Lafarge, has shown very similar results [4]: 92% of calcination degree and a calcination temperature increase of 60-70°C.

It is evident that also in full scale operation the increase of the calcination temperature will be associated with the oxyfuel mode. At this stage of investigation, how much this phenomena will affect the kinetics of the reaction taking place in the calciner volume is rather difficult to predict. The temperature interval between 840 °C and 1000 °C is characterized not only by the calcination but also by many initial steps of reactions involving Si, Al and Fe oxides, influenced by the presence of minor elements like Na, K, Cl. In particular, as soon as the temperature of 900 °C is passed, very small amount of liquid phase can appear and some new compounds are formed, like Gehlenite ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$), which decomposes at 1100 °C, and $12\text{CaO} \cdot 7\text{Al}_2\text{O}_3$, later on transformed in $3\text{CaO} \cdot \text{Al}_2\text{O}_3$, as soon as the material reach the temperature of 1100°C [5,6]. The early formation of these compounds in the calciner, due to the above mentioned increase of decarbonation temperature, together with the appearance of even limited amount of liquid phase, can cause unwanted coating at the calciner outlet. The temperature control at the outlet of the calciner is in the end of primary importance.

3 EVALUATION OF RESULTS OBTAINED IN TASK 8.3

3.1 Evaluation of the experimental environment

The first trial campaign put in evidence some limits in the in the initial test rig configuration. In particular it was necessary to go closer to the relevant industrial conditions in which a calciner normally operate; this means that both the raw material and the gas had to be preheated to get results in some way comparable with the full scale case. Other modifications to the original equipment have been introduced to better measure the temperature in the gas phase.

- The mechanism of thermal energy transfer and the measure of temperatures inside the reactor are critical issues: in the test rig the thermal energy is coming from the walls of the reactor, which are hotter than the gas and solid admixture, while, as already mentioned in the paragraph 1, in the real case the energy is coming from the diffuse combustion of the fuel and the wall temperature is in some way lower than that of the gas, solid fuel and raw mix admixture. Considering that the ratio surface/volume in a real calciner is much lower than that of the test rig, the heat transmission from the wall to the reactor core can be considered a good simulation of the transmission of heat from coal burning particles to the raw mix in suspension. In other words, from the point of view of the heat transfer mechanism, we can consider the test rig as a simulation of a volume inside the calciner.
- It was also clear during the tests execution that the original temperature measurements taken at the wall level were too high and not representative of the gas temperature: a Δt of about 40 °C has been measured. It is quite possible that also this value is not high enough because the thermocouple is influenced by the wall radiation. Once again this aspect is not far from the real case where the coal particles are burning at much higher temperature than that measured in the gas and solid phase.

In the end we can consider that the calcination tests have been performed in a way that goes rather close to the real industrial conditions.

While comparing the test results with the real case, another aspect that should be taken into account is that in the industrial practice the degree of calcination of the raw meal is overestimated. The main reason for this is that the raw mix that is treated in the calciner is not efficiently separated in the last cyclone (typically the fifth stage). At this level the efficiency is considered not higher than 90%, which means that about 10% of the calcined mix go back to the fourth stage and then again to the calciner, giving the impression that 15-20% of the raw mix enters the calciner already calcined, while less than only 5-6% was calcined before entering the calciner. The same can be considered for the calcined mix entering the rotary kiln after being captured in the fifth cyclone. The raw mix complete the calcination in the rotary kiln but part of it goes back to the fifth cyclone. Any sample taken at fifth cyclone exit is in this way overestimating the degree of calcination of the raw mix entering the rotary kiln. In practice the effective efficiency of the calciner doesn't change: instead of working between 20 and 95% of decarbonation it really works between 10 and 85%; in particular the amount of heat which is necessary to the calciner doesn't change. It has nevertheless some importance for the tests in discussion which are only apparently not reaching the same efficiency of the real calciner. An 85-90% of decarbonation degree as achieved in the tests can be considered an optimum and representative value.

Considering what was mentioned above, it is possible to conclude that the experimental conditions go rather close to the industrial conditions and are consequently able to produce results valid for future tests at higher TRL.

3.2 Test parameters

The tests had the objective to evaluate the degree of calcination of a real industrial raw meal obtained in two different operating conditions, air calcination and oxyfuel calcination, in reference to three main different operating parameters: temperature, residence time, and CO₂ concentration.

Table 3.2.1 Test parameters

Test	Temperature (°C)	CO ₂ concentration (vol.%)	Residence time (s)
Air calcination	780-920	20	3-9
Oxyfuel calcination	880-960	80	3-9

3.3 Test results

In the following paragraphs the test results are summarized adding the main comments and considerations.

3.3.1 Air vs Oxyfuel calcination

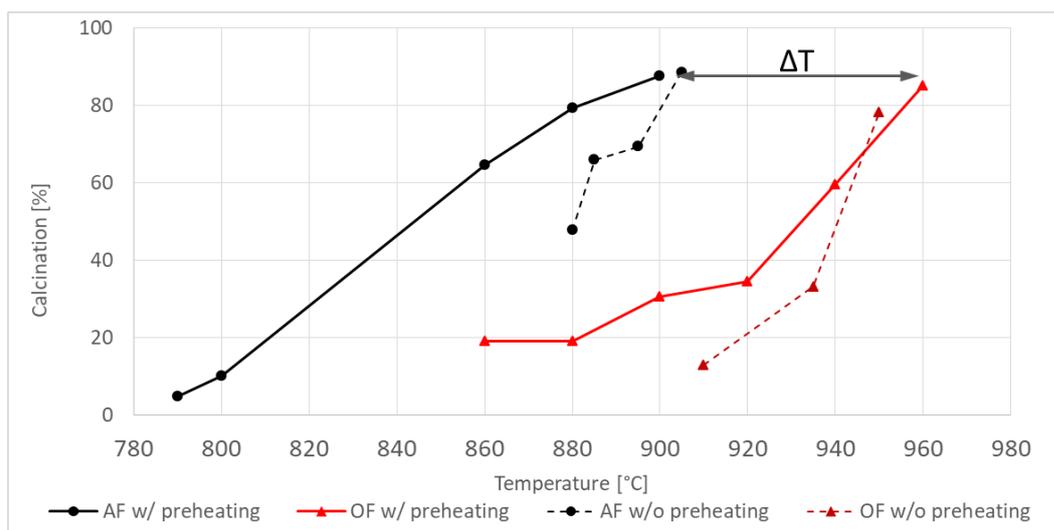


Figure 3.3.1.1: Air and oxyfuel calcination degree with and without preheating of gas and raw mix before feeding.

In an atmosphere containing 80% of CO₂, as in the case of oxyfuel calcination, the 90% of calcination degree is obtained only at a relatively high temperature; the Δt between air calcination and oxyfuel calcination is estimated in +60 °C, in good agreement with literature previous studies.

In line with what has already been said at the second point of paragraph 3.1, the absolute values of calcination temperature should be reduced by about 40 °C, respectively at 860 and 920 °C.

This is confirmed by Thermo-Gravimetric-Analysis (TGA) made on the raw meal samples in air and oxyfuel atmosphere.

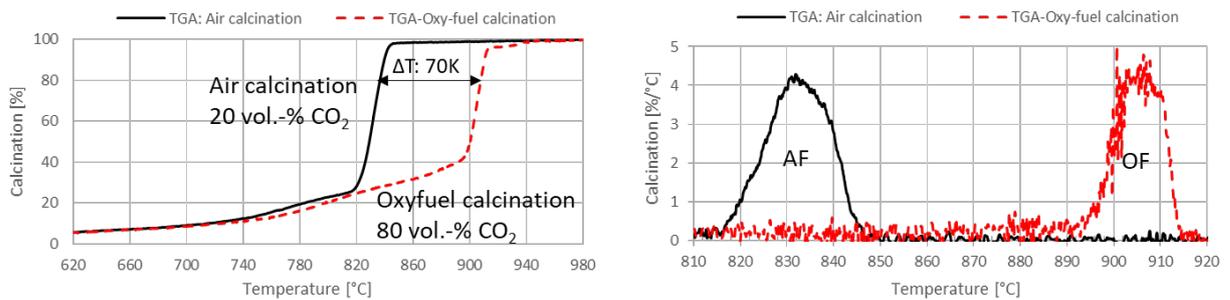


Figure 3.3.1.2: Air and oxyfuel calcination degree (left), calcination rate (right), from TGA tests

From the above figure it is evident that air calcination starts at 816 °C and is completed at 846 °C, while the same interval is shifted by 70 °C in case of oxyfuel calcination (894-914 °C). The peaks of decarbonation intensity (see the calcination rate diagram) are respectively 835 °C and 905 °C.

The temperatures about 844 °C measured in an industrial calciner using conventional fuels confirm the air calcination degree data reported by the first diagram of figure 3.3.1.2.

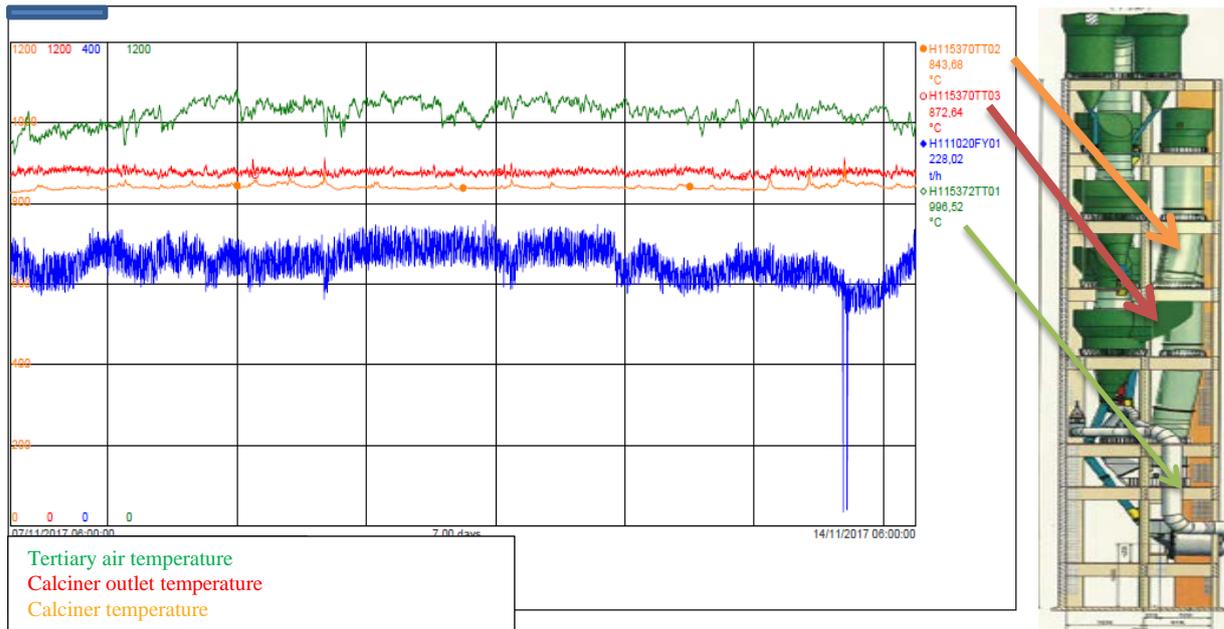


Figure 3.3.1.3: weekly temperatures and feeding rate trends in an in-line precalciner

3.3.2 Calcination vs. residence time

The calcination degree achieved at various temperatures is shown in fig. 3.3.2.1

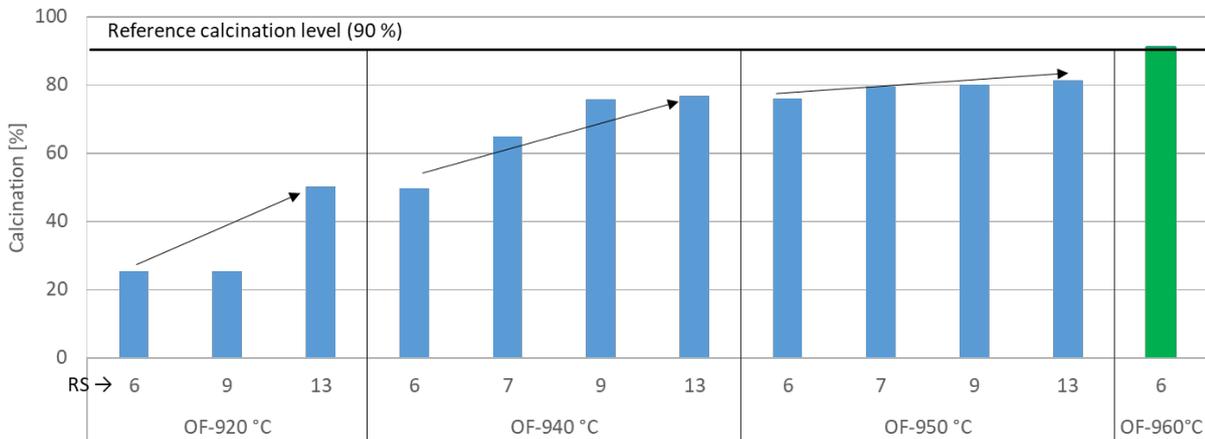


Figure 3.3.2.1: Residence time from the injection point to the sampling point (s)

The residence time (RS) variation from 6 to 13 s is even higher than the one normally practiced in industrial calciners (3-7 s). In any case to achieve a calcination level comparable with the reference level of 90% it is necessary to increase the reactor temperature to around 960 °C. This means that the real controlling factor is not the residence time but the temperature level.

3.3.3 Influence of CO₂ concentration: CO₂ concentration vs calcination temperature

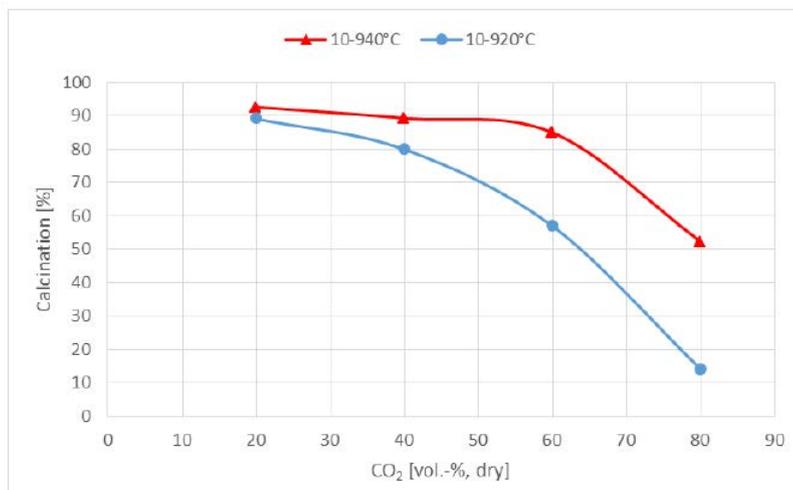


Figure 3.3.3.1: Calcination degree (%) at various CO₂ concentration

At two different temperatures the calcination degree has been controlled. In both cases the CO₂ concentration was increased from 20% vol, as in normal air calcination, up to 80% vol as for oxyfuel process conditions. A dramatic drop of calcination degree is more than evident mainly when the temperature is limited to 920 °C. Fairly better situation is observed at 940 °C even if, when the CO₂ concentration is around 80%, the calcination degree doesn't go further than 50%. These reported temperatures are not corrected for the effect of wall radiation as reported in paragraph 3.1 and 3.3.1.

3.3.4 Other verified impacts: particle size distribution and water vapor influence

Calcination tests have been repeated using always the same raw mix but selecting lower size particles, passing from the distribution of a normal raw meal (as received)

D ₁₀ 1.6 μm	D ₅₀ 8.9 μm	D ₉₀ 101.6 μm
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to a much finer granulometry (raw mix sieved at 50 μm)

D ₁₀ 1.5 μm	D ₅₀ 6.4 μm	D ₉₀ 30.4 μm
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As expected for finer raw meal the calcination degree shows an evident improvement, as it is well known also in normal practice, but the achieved calcination degree is not enough at temperature lower than 960 °C (Figure 3.3.4.1).

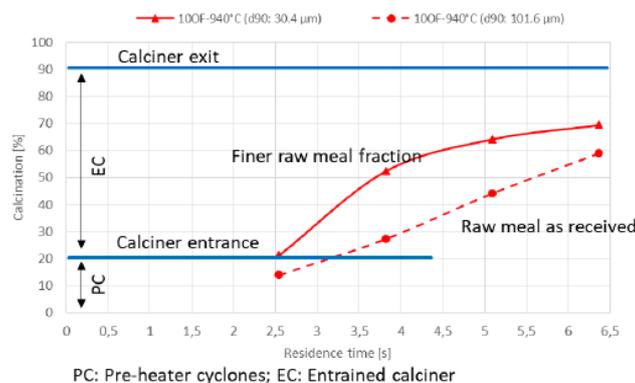


Figure 3.3.4.1: Calcination degree (%) of two different granulometries as a function of residence time

In the tests described up to this point the water vapor was not included in the gas composition, while it is always present in the real case, where it is deriving from combustion process and from dehydration of chemically bounded water in raw materials. To verify potential influences of water vapor on the calcination degree, water vapor (10% vol.) has been added at 940 °C at different residence time. The decarbonation degree is improved at the same level as it can be expected from the proportional decrease of concentration of CO₂. In practice no direct effect can be attributed to the presence of water vapor.

3.3.5 Test with fuel addition

The energy necessary for heating the raw meal and to supply the thermal energy requested by the calcium carbonate dissociation has been supplied up to this final phase only by the electrical energy used to heat up the wall of the reactor pipe. In addition, with this last series of tests, ground dosed fuel has been introduced (0,1 kg/h) in the reactor with the raw meal (1 kg/h). German lignite, 46.6 % in volatile matter, has been used as representative of fossil fuels, while ground torrified wood, a biomass 65% in volatile matter, has been used as alternative fuel. Accordingly with the higher content in volatiles, the biomass was ground at a coarser size than the lignite. To verify the calcination level of the raw meal all the tests have been repeated twice, one in normal industrial condition (20% of CO₂) and one in Oxyfuel condition (80% CO₂).

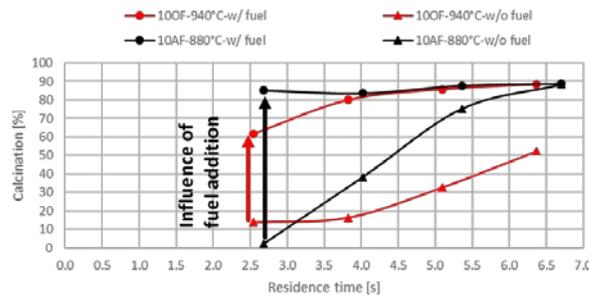


Figure 3.3.5.1 Comparison of results with (circles) and without (triangles) the use of lignite. Calcination degree at different residence time (black lines AF: air case for normal industrial condition with 20% of CO₂, red lines OF: oxyfuel case at 80% CO₂)

As expected the use of an additional source of thermal energy, not concentrated at periphery of the calciner but evenly distributed inside the raw meal mass, improve quite a lot the efficiency of the system: after only 2.5 seconds the calcination degree reach the value of 80% at 880°C (20% of CO₂), and 60% at 940°C (80%CO₂), against almost 0% and 12% respectively without added fuel. To go up to 100% of calcination degree further increase in calcination temperature should be necessary, probably not lower than 960°C in oxyfuel mode with fuel injection.

There is even more evidence of the beneficiary effect of fuel injection. In the following graphs of figure 3.3.5.2 the influence of temperature has been checked; of course it is positive in both cases of air combustion and oxyfuel combustion, but the calcination degree of 90% is reached at 880 and 940°C respectively, while without fuel injection it was necessary to reach the value of 900 and 960°C (see figure 3.3.1.1at page 6).

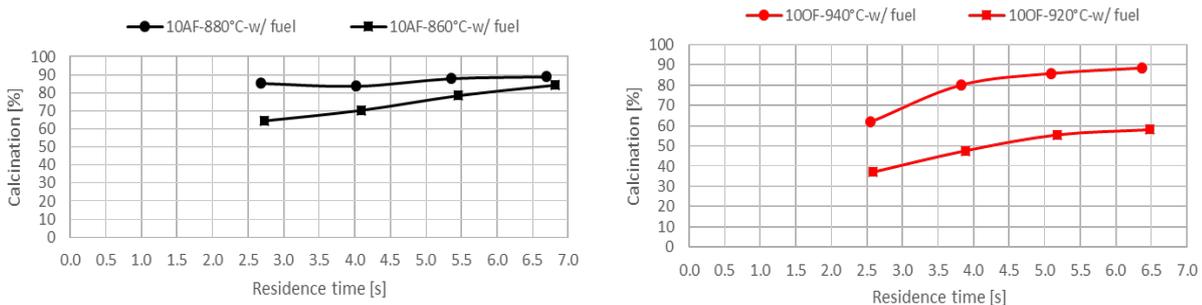


Figure 3.3.5.2 Calcination degree at different residence time with the same fuel at two different temperatures, left air case – right oxyfuel case

The use of a biomass fuel has also been investigated. The comparison between the two fuels is reported in the graph of figure 3.3.5.3. The amount of biomass burned in this test is higher than that of lignite to compensate proportionally the lower calorific value of the biomass. It is interesting to note that, all the operative conditions being the same, the degree of calcination after 2.5 seconds is significantly higher when the biomass fuel is used. This is most probably due to the higher content in volatiles which allows for an earlier start of the burning process with a quicker release of thermal energy in the biomass case. Both fuels reach the maximum of calcination degree after 6.5 seconds.

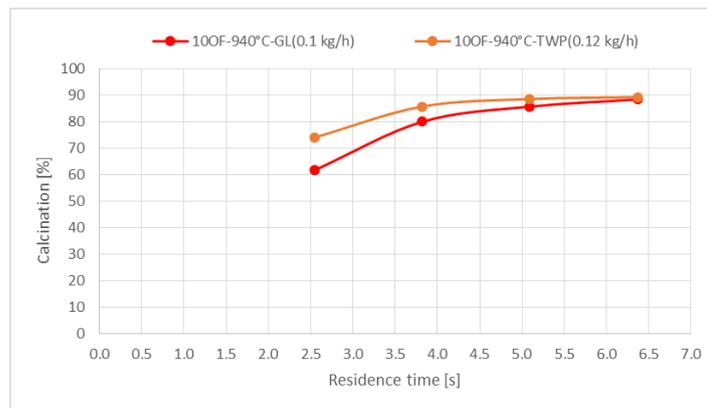


Figure 3.3.5.3 Calcination degree at different residence time using two different fuels

4 PARAMETERS AND FINDINGS FOR EXPERIMENTS TO SUPPORT THE DESIGN OF AN OXYFUEL CALCINERS

The calcination in oxyfuel mode has been explored almost completely in relation to a number of influencing factors and for each of them a sufficiently wide range of variation has been considered. The effects on the decarbonation reaction efficiency have been evaluated and coherent results obtained. Nevertheless the way to the oxyfuel technology application is still very long.

The main information collected in this test campaign are that, to obtain an high degree of decarbonation, it is necessary to increase in non-negligible amount the temperature inside the calciner and that all the other possible influencing factors, including the residence time, are not as important as the temperature level to reach a calcination degree close to the one industrially obtained with the current technology. It is necessary to remember that it is very important to obtain in oxyfuel mode almost the same calcination degree because this opens the door to the adoption of the oxyfuel technology by the existing cement kilns. Otherwise the oxyfuel technology would only be applicable to new lines, which especially in Europe are foreseen as very few in next decades. The use of a fuel, fossil or non fossil, seems to mitigate this effect initially evaluated close to 70°C and in these conditions, much closer to the real one realized in an industrial calciner probably lower than 50°C.

The temperature increase of 50°C has no relevant effects on the sizing of the calciner and not even on the refractory design but still may have important and negative consequences on the process chemistry, as described at the end of paragraph 3. Coating formation inside the kiln is a positive effect as soon as it produces a sort of protection of the refractory lining, installed in various forms and kinds, all over the internal surfaces of the kiln, from the cooler to the top of the preheater tower. On the other hand the formation of thick coating layers is a very negative phenomenon which jeopardizes the production levels and in many cases is cause of problems in the production process and kiln stoppages.

In an oxyfuel kiln this problem is even more severe if we consider that it is not possible to destroy the coating with mechanical actions, opening doors or manhole to have direct access to the zone to be cleaned, as it is normal practice in conventional operating mode. Air leakage is already a problem even in a very tight kiln operated in oxyfuel mode and the opening of a door will provoke unacceptable dilution of the CO₂ recovered by inflow of air. To verify the real impact of this coating formation on the future oxyfuel kilns it is necessary to operate continuously in oxyfuel mode for a rather long time, at least in the order of a few days, because this is an accumulation phenomenon that takes relatively long time to create the build-up and the related negative effects (high pressure drop, reduced/irregular flow of raw material, insufficient oxygen level at the burning zones).

Also the kind of raw materials used can contribute to increase the impact of anomalous coating formation. The presence of high levels of Sulphur, Chlorine, Alkalines is since ever known as a potential source of troubles even in conventional operations. Kiln operators know how to handle these problems but to a certain limit only.

The residence time and the fineness of raw materials don't have an impact on calcination degree as important as that of temperature, but their increase is clearly effecting in a positive way the calcination degree and could supply a possible solution, if the coating problem arises.

Since the day the alternative fuels started to be used in the cement kiln process, to substitute more expensive fossil fuels, the need to increase the residence time and consequently the volume of the calciner has been felt. During the last 15-20 years new lines and many of the existing ones in Europe have been equipped with calciners, having, at nominal production, a retention time of 6-7 s. It seems that these kilns are already equipped for the possibility of managing the calcination for clinker production in oxyfuel mode. Quite frequently, if not always, the increase of the calciner volume is, technically speaking, not very challenging, because what has to be changed is not the section of the duct, which changes the footprint of the calciner, but only the length, a modification that is in general possible getting out with the duct from the existing tower structure, providing additional external supports standing on ground level.

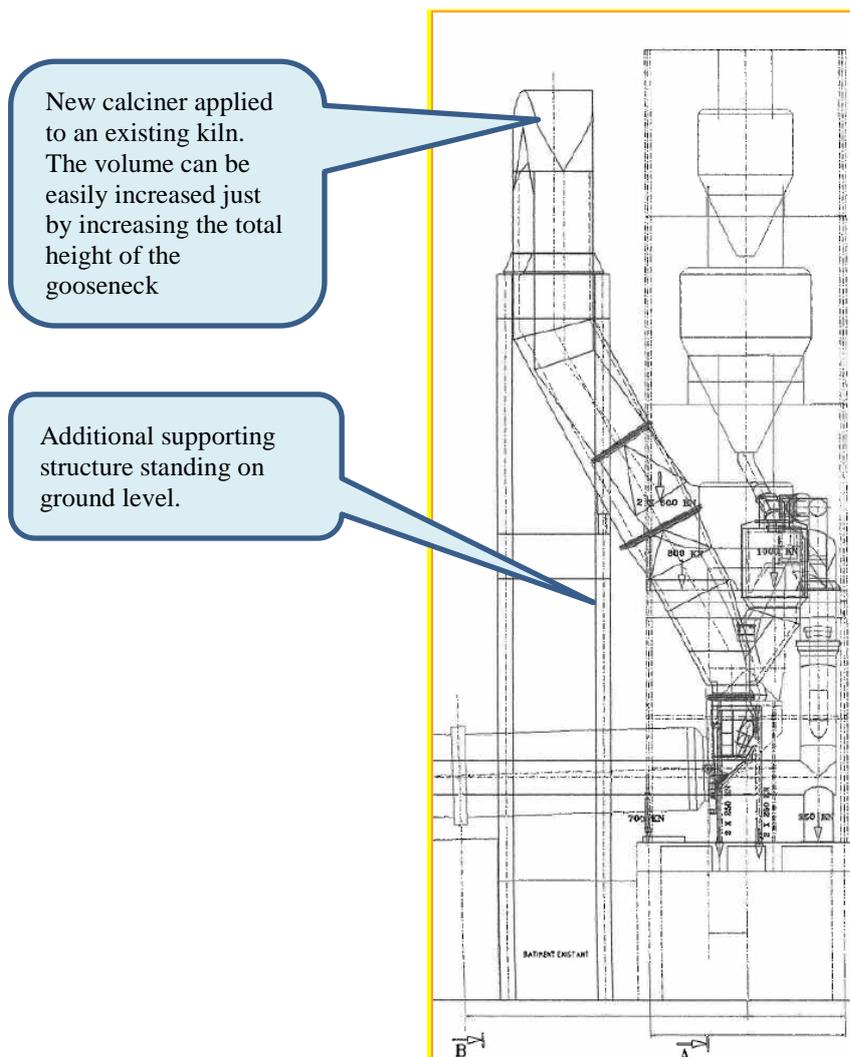


Figure 4.1: Example of a project for the implementation of an in-line calciner to the preheater of an existing cement kiln.

5 CONCLUSIONS

The work done in the frame of WP8 has been very useful to provide the necessary information for the design and operation parameters of an oxyfuel calciner to WP6. In particular it is confirmed that the in-line configuration is the most suitable, is the simplest one and consequently less exposed to the risk of false air leakage. From the process point of view, according to the results obtained during the tests, it is clear that in oxyfuel mode the calcination temperature, as by the way expected from the theory, shows an evident increase in the range of 40-60°C, probably higher than expected; final conclusions on the effective increase must be derived from tests operated in an industrial environment (TRL8).

Other process parameters have been investigated, as retention time, CO₂ concentration, particle size distribution, and all of them have been demonstrated as not able to produce a satisfactory level of calcination (close to 90%) if the operating temperature is not high enough (between 940 and 960 °C). This level of increase in the calcination temperature introduces some concern about possible formation of unwanted coating at the calciner outlet, for the premature appearance of liquid phase and of clinkerization reactions. The importance of these phenomena can't be evaluated in the present test rig and further investigations are necessary at size of installations where the oxyfuel mode can be operated for a reasonable period of time (order of days).

All the results obtained are coherent with what is reported in the literature and within themselves, notwithstanding all the technical problems that have been necessary to solve in order to reproduce, at a relatively small scale, a complex environment and to measure temperatures and flow rates in difficult test conditions.

6 REFERENCES

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