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## A twin-bed test reactor for characterization of calcium looping sorbents

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### ABSTRACT

The loss of sorbent CO<sub>2</sub> capture capacity and the extent of particle attrition over iterated cycles are relevant to the design of calcium looping processes. Thermogravimetric analyzers or single batch fluidized bed reactors are typically used at the lab-scale to evaluate the sorbent performance. One drawback of these devices is that they do not reproduce the thermal history that is actually experienced by sorbent particles in real looping cycles. In this study, a novel experimental device is proposed to overcome this limitation. The apparatus consists of two interconnected batch fluidized bed reactors operating as calciner and carbonator, respectively. The two reactors are connected to each other by a duct (whose openings can be located at an adjustable level above the gas distributor) which permits rapid and complete pneumatic transport of the sorbent (limestone) between the reactors.

Preliminary tests at ambient temperature were first performed to find the best operating conditions in terms of limestone conveying efficiency. Under the optimal experimental conditions >95% collection efficiency of the limestone was obtained.

High-temperature tests were carried out to simulate multiple calcination/carbonation cycles. The results indicate that the device is able to effectively transfer the sorbent between the two reactors while keeping the temperature variations limited.

The test reactor has been used to assess the effect of the thermal history of the limestone on its CO<sub>2</sub> capture capacity and attrition tendency. The results were compared to those previously obtained with the same limestone under comparable operating conditions in a single bed apparatus. The comparison reveals that the thermal history experienced by the limestone has a non-negligible effect on its performance.

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### 1. Introduction

Calcium looping (Ca-L) is a promising technology which uses Ca-based sorbents, such as limestone or dolomite, to capture CO<sub>2</sub> contained in power plant flue gas [1–10]. One of the main advantages of this technology is the utilization of cheap, natural and non-toxic sorbent materials which entail a competitive cost for CO<sub>2</sub> capture as compared to other proposed techniques. Moreover, Ca-L, being a post-combustion technology, could be used to retrofit existing power plants.

The Ca-L process is based on the use of two reactors, the carbonator and the calciner, which typically consist of two interconnected fast fluidized beds [4,11]. The fluidized bed (FB) technology allows easy sorbent circulation between the two reactors and assures a good contact between the gas and solid phases. In the carbonator the lime-based sorbent captures the CO<sub>2</sub> coming from the power plant, purifying the exhaust gas and producing a carbonated sorbent. The typical temperature in the carbonator is in the range 650–700 °C. Subsequently, the carbonated sorbent is transferred to the calciner where calcination occurs at a higher

temperature (850–950 °C) regenerating the calcium oxide, which is thus ready for a new cycle, and producing an almost pure CO<sub>2</sub> stream, ready for further purification, compression and final transportation to the storage site.

However, the Ca-L technology is affected by two limitations which may partly compromise its industrial application. The first one is related to the strong sorbent deactivation with the number of consecutive cycles. This deactivation is mainly due to high-temperature sorbent sintering, which decreases the sorbent pore area, and to the possible presence of SO<sub>2</sub> in the flue gas, which irreversibly reacts with lime producing calcium sulfate [12–23]. These two processes together determine a progressive dramatic reduction of the CO<sub>2</sub> capture capacity of the sorbent. The second problem is connected to the reactor configuration: the use of FB reactors implies the occurrence of sorbent attrition due to particle impacts [24]. This phenomenon causes the change of the sorbent particle size distribution in the bed and produces fine fragments which are elutriated with the outgoing gas stream, determining a net sorbent loss from the system.

In the last years, many efforts have been addressed to evaluate the effects of the CO<sub>2</sub> capture capacity decay and of attrition on the Ca-L system, and if it is possible to overcome or at least to limit these effects. The

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typical devices used for routine sorbent characterization are thermogravimetric (TG) analyzers and single lab-scale fluidized beds, which have the advantage of being handy [25]. Unfortunately, these techniques are affected by several limitations. For example, TG tests do not permit evaluating the attrition propensity of the sorbent. Moreover, in both TG and single FB tests it is not possible to simulate a continuous Ca-L process where the sorbent particles are cyclically transferred from one reactor to the other. Hence, the real thermal shock conditions, which the sorbent undergoes, cannot be reproduced. This limitation has important consequences because the thermal history of the sorbent may have a strong influence on both its CO<sub>2</sub> capture capacity and its attrition propensity. On the other hand, the scaling-down of a continuous calcium looping dual fluidized bed apparatus to the lab-scale is particularly complex and involves many operational difficulties.

In order to overcome these limitations, a novel batch lab-scale apparatus has been recently presented, which is suited to routinely characterize at the lab-scale the performance of sorbents in a Ca-L process approaching a realistic sorbent particle thermal history [26]. This system consists of two twin lab-scale bubbling fluidized beds working separately in a batch mode, which are connected to each other by a duct for the fast conveying of the sorbent between the reactors. Noteworthy, the apparatus is not meant to simulate a continuous calcium looping process, but only to reproduce a sorbent particle thermal history as close as possible to reality, in terms of cycling of temperatures and of reaction atmospheres. In this study we report the first experimental results of a limestone sorbent characterization, where the novel apparatus has been applied under both ambient and high-temperature conditions. The hot-bed results have been also compared to similar tests carried out in a single FB apparatus on the same limestone, in order to highlight possible effects of the thermal history on the sorbent performance.

## 2. Experimental

Fig. 1 shows the concept of the lab-scale apparatus employed in this work. The system consists of two identical fluidized beds, where one reactor is operated as the calciner and the other one as the carbonator. A pipeline (transport duct), with a diameter smaller than the reactor size, is lowered in both reactors and partly immersed into the bed material. Operation of the valves located on the duct and at the top gas exit of both reactors allows conveying the sorbent from the calciner to the carbonator and vice-versa. In particular, during a calcination (or carbonation) stage the valves on the duct are closed, while the valves on the top gas exit are open. When the calcination stage is over, the top gas exit valve of the calciner is closed and the duct valves are opened to permit the fast pneumatic transport of the material in the other reactor. In this way, the sorbent undergoes the typical thermal shock of a Ca-L system. An important feature of the test procedure is that silica sand is used as a buffering material in both reactors in order to avoid significant bed temperature variations due to chemical reactions and conveying of

solids. This latter point highlights a crucial issue since the system must be designed to permit the quantitative pneumatic transport of the sorbent while limiting the sand transportation.

For this reason, a preliminary experimental campaign was carried out as a proof-of-concept for the system, using a lab-scale fluidized bed made of plexiglass [26]. Eventually, an apparatus consisting of two stainless steel fluidized beds (twin-beds) equipped with a conveying system (Fig. 2) was designed and built up on the basis of the experimental data produced by the plexiglass FB. The bed on the right was called calciner bed (Cal-B), while the other carbonator bed (Car-B). The two reactors have an inner diameter of 40 mm and are equipped with a stainless steel connecting duct with an inner diameter of 10 mm. Each reactor consists of three sections: a wind box (0.66 m) which can be also used as a preheater zone; a fluidized bed (1.0 m) which is separated from the wind box by a perforated plate with 55 holes of 0.5 mm diameter in a triangular pitch; and finally at the top a gas exit duct leading to a two-exit valve with two compartments to house sintered steel filters for elutriated fines collection (whose filtration efficiency is >99% for >10 μm particles). A steel hopper for solid feeding to the bed is connected sideways to each reactor. The apparatus is equipped with an additional duct, located in the middle between the twin beds and connected with the transport duct by means of a 3-way valve. At the bottom of this duct a removable stainless steel container is placed. The aim of this duct is to carry out a fast discharge of the reactors and to collect the whole bed material into the container. All gases were fed to the reactors by high-precision mass flowmeters/controllers.

For all the tests, silica sand in the size range of 900–1000 μm ( $U_{mf} = 0.3$  m/s,  $U_t = 7.8$  m/s @ 850 °C) was used as buffering bed material, while a German high-calcium (CaCO<sub>3</sub> > 99%) limestone (EnBW, particle size range 400–600 μm,  $U_{mf} = 0.06$  m/s,  $U_t = 3.6$  m/s @ 850 °C) was used as sorbent material. The size difference between the two materials permits to easily separate them after the tests by sieving. In addition, this size difference determines during the fluidized bed operation a segregation of the smaller and lighter sorbent material to the top section of the bed and that of the larger and heavier sand material to the lower section of the bed. This partial separation of the two materials in the bed can be exploited to obtain preferential transport of the sorbent with respect to the sand, by carefully tuning the penetration depth of the transport duct in the bed [26–28].

A first set of tests at ambient temperature was carried out by varying the distance between the immersed duct and the plate distributor and at different fluidization velocities (see Table 1), according to the following procedure: the fluidized bed was filled with different amounts of silica sand and limestone and fluidized by a flow of air under bubbling conditions. Table 1 reports the conditions used for the tests:  $v_f$  indicates the fluidization velocity,  $h_D$  is the distance between the duct and the plate distributor, while  $h_b$  is the difference between the static height of the whole bed (sand + limestone) and  $h_D$ , thus representing the immersion depth of the duct inside the bed. In all the tests 20 g of limestone and 150 g of sand were used, representing relative contributions to the static bed height of 11 and 76 mm, respectively. During the initial stage the duct valve was closed while the bed gas exit valve was open. After about 15 min of operation (a time long enough to achieve steady segregation conditions in the bed [26]) the duct valve was opened and the bed valve was closed simultaneously. In these conditions all the gas flow passed through the duct and the solid pneumatic transport occurred (being the gas velocity in the duct larger than the particle terminal velocity). The material was collected in a bin and then sieved to separate the limestone from the sand using a 710 μm-sieve. Finally, both limestone and sand were weighted to calculate the transport efficiency of the two materials. The transport efficiency  $\eta$  is defined as the ratio between the amount of the transported material and the total amount initially charged in the bed. The aim of these tests was to understand the influence of  $v_f$  and  $h_b$  on the transport efficiency in order to find the best conditions to maximize limestone transport and minimize sand transport.

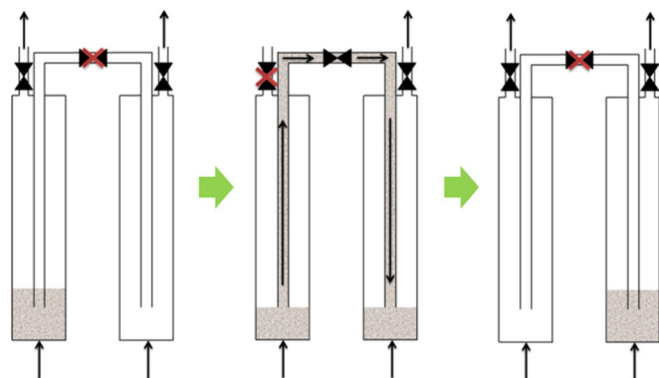


Fig. 1. Scheme of the solid transport procedure between the two fluidized bed reactors.

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