

Enhancement of CaO for CO₂ capture in an FBC environment

C. Salvador^a, D. Lu^a, E.J. Anthony^{a,*}, J.C. Abanades^b

^a CANMET Energy Technology Centre, Natural Resources Canada, 1 Haanel Drive, Ottawa, Ont., Canada K1A 1M1

^b Department of Energy and Environment, Instituto de Carboquímica (CSIC), Miguel Luesma 4, 50015 Zaragoza, Spain

Abstract

Experiments have been carried out on three Canadian limestones to determine their ability to remove CO₂ in multiple carbonation/calcination cycles. Two systems have been used: a circulating fluidized bed combustor (CFBC) operated in the bubbling FBC mode; and a thermogravimetric analyzer (TGA). The falloff in CO₂ capture ability of the limes derived from these limestones was initially in agreement with an empirical correlation, but subsequently the decay in performance was slower. The use of Na₂CO₃ and NaCl to reactivate the lime and enhance CO₂ capture failed to do so in the FBC environment, but in the case of NaCl, produced significant improvements in performance in the TGA after several cycles, while Na₂CO₃ did not improve performance in either case. The use of 100% CO₂ failed totally to reactivate sorbents in the TGA, but did improve performance in the FBC. There is also evidence from surface area measurements that carbonation in 100% CO₂ atmospheres influences pore size and surface area in the FBC environment. These results suggest that 100% CO₂ atmospheres may provide a possible reactivation method for some limestones and that the use of NaCl and Na₂CO₃ for this purpose requires further investigation in FBC environment.

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1. Introduction

Fossil fuel combustion systems such as coal-fired power plants are one of the major sources of CO₂ emissions, the major contributor of greenhouse gas (GHG) concentration in the atmosphere. Increasing atmospheric CO₂ concentration and concern over its effect on global warming is a powerful driving force for the development of new advanced energy cycles incorporating CO₂ capture. A potential approach to reducing CO₂ emissions is the separation of CO₂ from flue gases from conventional air-blown combustion systems and storage of CO₂ in underground geological formations (coal beds, oil reservoirs, deep saline aquifers) or in the deep ocean.

Numerous CO₂ separation processes are currently being tested for deployment in fossil-fuel-based power plants. One method to burn coal and produce high-CO₂ flue gas (>95%) is called the “O₂/CO₂ combustion process”, which is considered to be a most energy-efficient process [1]. However, significant energy consumption in separating O₂ from air and recycling flue gas adversely affect the economics of such approaches. Attempts have been made to separate CO₂ from flue gas using absorption by amine solution and adsorption

by solids such as zeolites [2]. Absorption processes employ physical and chemical solvents such as Selexol and Rectisol [3]. Adsorption systems capture CO₂ on a bed of adsorbent materials such as molecular sieves or activated carbon [4]. CO₂ can also be separated from flue gases by condensing it out at cryogenic temperatures [2]. Polymers, metals such as palladium, and molecular sieves are also being evaluated for membrane-based separation processes [2]. It is generally accepted that the cost associated with the separation of CO₂ from flue gases introduces the largest economic penalty to these mitigation options [1,2]. This justifies development of a range of emerging approaches to separate CO₂ by more cost-effective processes.

The possibility of using the carbonation reaction for the removal of CO₂ from a gas stream was already considered in the late 19th century. Recently however, it has been suggested that calcined limestones may be able to remove CO₂ in the fluidized bed combustion environment and, by subsequent calcination, produce a pure CO₂ stream for sequestration, in a process based on CO₂ chemical looping [5,6]. This scheme involves the use of: a pressurized fluidized bed combustor/carbonator (PFBC/C) where the fuel is burned in an excess of lime which, depending on operating conditions, can remove up to 80% or more of the CO₂ and effectively all of the SO₂; and a calciner where sorbent is regenerated by burning minor proportions of the fuel in O₂. The pure

* Corresponding author. Tel.: +1-613-996-2868; fax: +1-613-992-9335.
E-mail address: banthony@nrcan.gc.ca (E.J. Anthony).

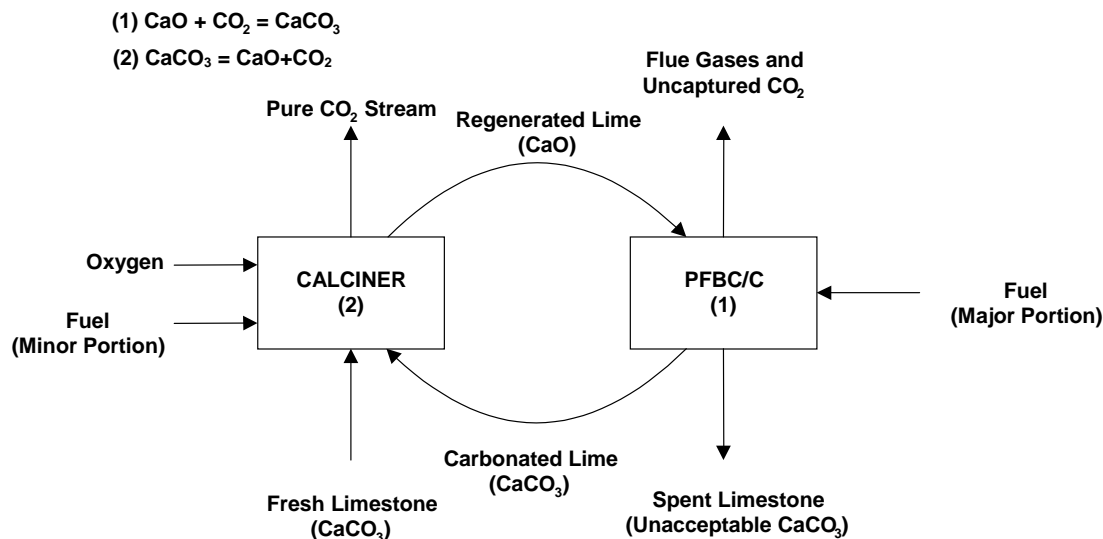


Fig. 1. CO_2 hot gas scrubbing concept (PFBC/C is the pressurized fluidized bed combustor and carbonator unit).

CO_2 emitted is either used for some purpose or sequestered (Fig. 1).

Such a process requires the lime-based sorbents to be recycled many times to reduce the sorbent make up flow. However, several studies on the reversibility of the carbonation and calcination reaction have shown that the recarbonation is far from reversible in practice [7,9–13]. After a rapid initial reaction period, controlled by the surface reaction resistance, a much slower second stage controlled by product layer diffusion follows. The difficult completion of recarbonation can be explained by considering structural property changes in the process of the reaction. Mess et al. [14] investigated the product layer diffusion during the reaction of single crystal lumps of pure CaO and found that the slow reaction period is associated with the build up of a thin CaCO_3 product layer (in the order of 100 nm). The progress of the carbonation reaction is negligible from that point at temperatures, partial pressures of CO_2 , and particle residence times relevant for a PFBC/C. Furthermore, the maximum carbonation capacity decreases rapidly with multiple cycles as a result of the loss of suitable pore volume in the lime-based sorbent during every calcination step [7]. This paper looks at pretreatment of limestones for CO_2 removal using salts and other methods of reactivation as a means of preventing or delaying this degradation of the texture of the sorbent or as a means of enhancing the mechanism of reaction during the slow reaction period.

2. Experimental work

The experimental work described below is first concerned with verification in FBC environment of the observations made by Abanades and Alvarez [7]; namely, that the maximum carbonation capacity is strictly a function of the num-

ber of calcination/carbonation cycles. In addition, this work also examined several sorbent reactivation strategies, in particular, the use of Na_2CO_3 and NaCl additives and pure CO_2 as means of reactivating lime. Experiments for both studies were carried out in a TGA and a FBC. A morphological study of samples taken from the FBC CO_2 reactivation experiments was also performed in order to gain further insights into the deactivation of the sorbents.

2.1. TGA—apparatus and methodology

A simplified schematic of the TGA is shown in Fig. 2. The TGA consists of an electronic balance (Cahn 1100), a vertical electric furnace, a reactor tube, carrier gas system and a computerized data acquisition system. The reactor tube is made of Inconel 600 alloy and has an inside diameter of 24 mm and a height of 900 mm. The reactor tube can be unscrewed from the TGA revealing a platinum sample holder (10 mm in diameter, 1.5 mm in depth). The electric furnace surrounds the reactor tube and is the primary heat source. The carrier gas system consists of a digital mass flow controller (Matheson Gas Products). Losses or gains in mass are measured by the balance and recorded by the data acquisition system. Changes in gas composition are also measured and recorded.

Limestone types tested included Havelock from eastern Canada, Cadomin from western Canada, and Kelly Rock from Nova Scotia, Canada. The chemical compositions of the limestones are given in Table 1. A summary of the experimental conditions for the TGA work is given in Table 2. Samples of 22–23 mg were placed in the reactor where they were calcined at 850°C and at atmospheric pressure in nitrogen, and then carbonated at 700°C and 1 bar in 15% CO_2 /85% N_2 gas mixture. A thermocouple was used to measure temperature just below the sample

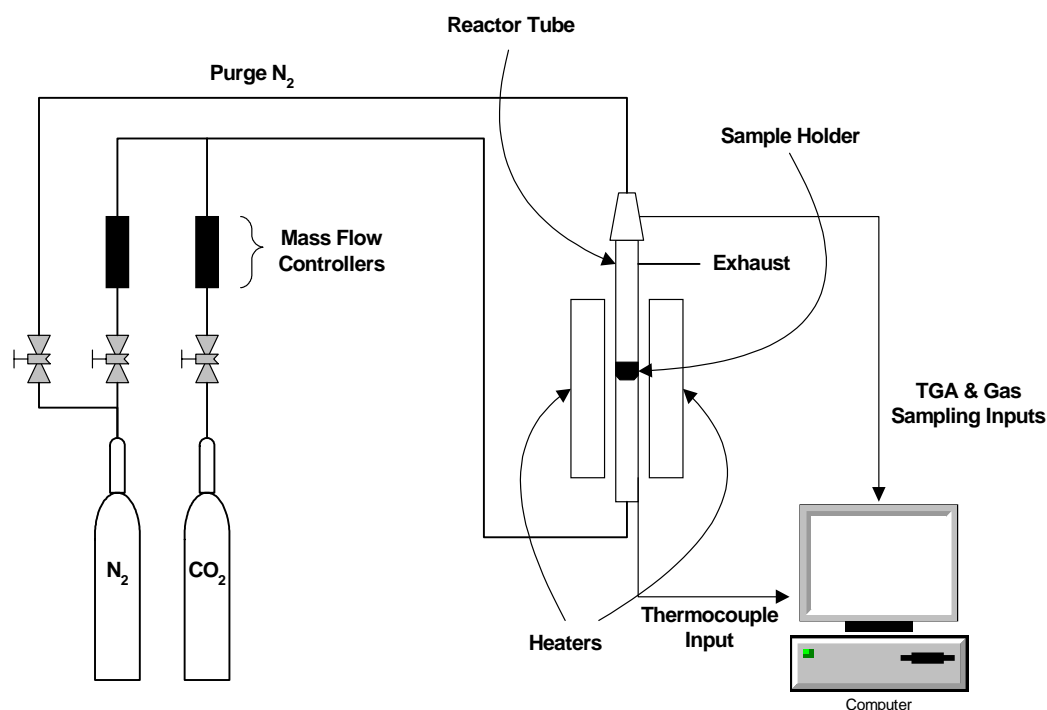


Fig. 2. Simplified schematic diagram of TGA equipment.

Table 1
Limestone composition, wt.%

Component	Havelock	Cadomin	Kelly rock
SiO ₂	1.23	1.50	5.31
Al ₂ O ₃	<0.38	<0.38	1.54
Fe ₂ O ₃	<0.55	<0.55	0.36
TiO ₂	<0.04	<0.04	0.08
P ₂ O ₅	<0.02	<0.02	<0.02
CaO	53.99	55.12	51.74
MgO	0.59	2.25	0.58
SO ₃	0.20	0.32	0.98
Na ₂ O	<0.17	<0.17	0.07
K ₂ O	<0.08	0.21	0.36
BaO	0.02	<0.02	0.18
SrO	0.02	<0.02	0.04
V ₂ O ₅	<0.02	<0.02	–
NiO	0.01	<0.01	–
MnO	0.08	–	0.16
Cr ₂ O ₃	<0.01	<0.01	–
LOF	43.34	42.77	43.14
SUM	99.81	102.19	104.55

Table 2
TGA experimental conditions

Limestone type	Havelock	Cadomin	Kelly Rock
Sample mass (mg)	22–23	22–23	22–23
Calcination temperature (°C)	850	850	850
Carbonation temperature (°C)	700	700	700
CO ₂ concentration in N ₂ (%)	15	15	15
CO ₂ concentration (%) (reactivation study)	100 (cycles 7 and 10)	100 (cycle 9)	NA
Na ₂ CO ₃ concentration per mole of CaCO ₃ (%) (reactivation study)	0.5–3 (all cycles)	NA	NA
NaCl concentration per mole of CaCO ₃ (%) (reactivation study)	0.5 (all cycles)	NA	NA
Particle size, D _p (μm)	650–1675	650–1675	650–1675

holder. The temperature and sample mass were recorded with respect to time in 5 s intervals until termination of the run.

As part of the investigation into the effects of salts on limestone carbonation in the TGA study, Havelock limestone was chosen as the sorbent for doping experiments with NaCl and Na₂CO₃. This limestone was also used in the work in the pilot-scale CFBC unit to investigate carbonation characteristics. The salts were prepared as 20 wt.% aqueous NaCl and Na₂CO₃ solutions, which were then poured over prescreened limestone samples with a particle size range of 650–1675 μm. The amounts of solution used in TGA investigations were equivalent to 0.5, 1, 2 or 3 wt.% of salt in limestone. The wet mixture was blended for 20 min and dried overnight at 120 °C. The samples were calcined at 850 °C in the TGA under 100 ml/min N₂ before carbonation runs. The CO₂ capture capacity of the sample was determined by weight gain over a period of 20 min.

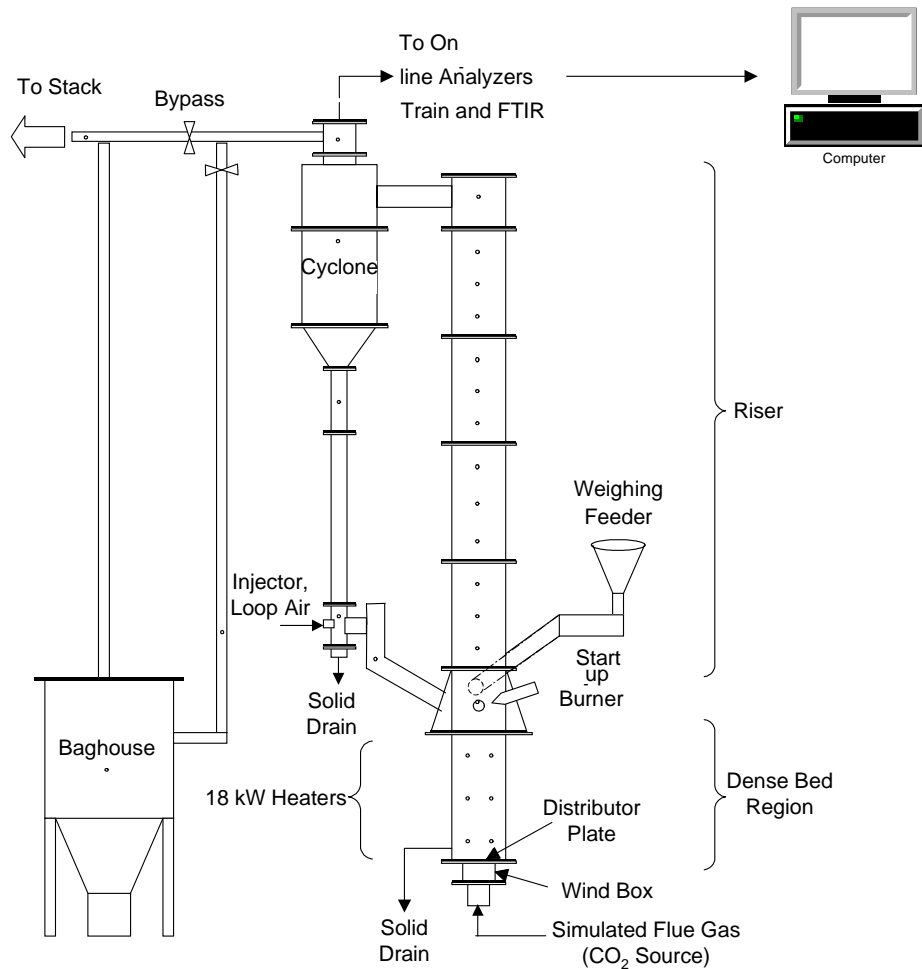


Fig. 3. Schematic diagram of pilot-scale FBC unit.

2.2. FBC—apparatus and methodology

The major components of the pilot-scale CFBC, used herein in the bubbling mode, consist of a dense bed region, riser section, cyclone and baghouse. A schematic of the FBC is shown in Fig. 3. The dense bed region is 1 m high with an internal diameter of 0.1 m. The combustion chamber section is surrounded by four electric heaters (18 kW total), which can provide supplemental heat during operation. The heaters

can maintain the dense bed region at temperatures of up to 900 °C. The unit is equipped with a data acquisition system which records temperature, pressure drop and gas composition.

Experimental conditions for the FBC work are listed in Table 3. Approximately 5 kg of limestone was used per experiment. Prior to the start of any experiment the limestone was sieved to ensure that particle size was between 650 and 1675 μm . The CFBC was operated as a bubbling fluidized

Table 3
FBC experimental conditions

Limestone type	Cadomin	Havelock
Initial bed mass (kg)	5	5
Fluidizing velocity (m/s)	1	1
Calcination temperature (°C)	850	850
Carbonation temperature (°C)	700	700
CO ₂ concentration (%)	15	15
CO ₂ concentration (%) (reactivation study)	100 (cycle 8)	100 (cycles 12 and 13)
Na ₂ CO ₃ concentration per mole of CaCO ₃ (%) (reactivation study)	NA	4 (all cycles)
NaCl concentration per mole of CaCO ₃ (%) (reactivation study)	NA	0.5 (all cycles)
Particle size, D_p (μm)	650–1675	650–1675

bed with a fluidizing velocity of 1 m/s during these tests to maintain control over the number of cycles experienced by particles. Limestone was calcined at 850 °C in air. Once the limestone was fully calcined the temperature in the bed was lowered to 700 °C and the lime was exposed to a mixture of air and CO₂ (CO₂ concentration was verified by direct measurement at the inlet of the dense bed region). The typical inlet CO₂ concentration was 15% for all tests except CO₂ reactivation tests where CaO was exposed to 100% CO₂ (see description below). The end of carbonation, which was evidenced by a rapid increase in CO₂ concentration at the exit of the system, marked the end of a cycle. The bed temperature was then increased back to 850 °C in preparation for a new calcination/carbonation cycle. Samples were collected periodically during the calcination and carbonation steps and tested to ensure complete calcination/carbonation was occurring. CO₂ capture capacity was determined from the difference in the CO₂ concentrations between the inlet and exit, the time before the rapid increase of CO₂ concentration occurred, and the amount of limestone.

CO₂ reactivation tests involved exposing the calcined limestone to pure CO₂ for 1 or 2 cycles at or near the end of a run, where an experimental run consists of between 8 and 14 cycles. Once carbonation was deemed complete, the limestone was calcined as described above. CO₂ reactivation experiments were performed on both Cadomin and Havelock limestones.

Two additional reactivation experiments were carried out on the Havelock limestone, exclusively. These experiments involved doping the limestone with 4% Na₂CO₃ per mole of CaCO₃ and 0.5% NaCl per mole of CaCO₃. The limestone was soaked in a solution of the additive (Na₂CO₃ or NaCl) and water for a period of 24 h. Water was slowly evaporated in an oven at 100 °C and atmospheric pressure. One 3-cycle

run was performed for each additive. The concentration of Na₂CO₃ and NaCl chosen is partially based on work by Razbin et al. [8].

2.3. Morphological study

For the FBC CO₂ reactivation experiments, cycles 11, 12 and 14 of the Havelock test, samples were collected for detailed microscopic examination. Carbonated lime samples collected at the end of each cycle were divided in two, half of which was calcined in an oven at 900 °C. Brunauer–Emmet–Teller (BET) surface area measurements were made for carbonated and calcined samples in addition to a scanning electron microscope (SEM) study. The results were compared with a sample of the parent limestone (initial bed material), which was also similarly examined.

A Hitachi Model 570 SEM was used to examine these samples. Two types of observations were made—surface observations, where particles are glued to a surface, and cross-section observations, where particles are embedded in resin, the sample cut and the surface polished. Photographs were obtained at magnifications of 40×, 200×, 1000× and 5000× for both sets of observations. BET surface area measurement of the particles was made using a Micromeritics ASAP 2000, which also provides information on the pore volume and average pore size.

3. Results and discussion

A typical raw process record of the weight–temperature–time data collected by the TGA for Cadomin limestone (11 calcination/carbonation cycles) is illustrated in Fig. 4. Complete calcination was achieved in each cycle, with the

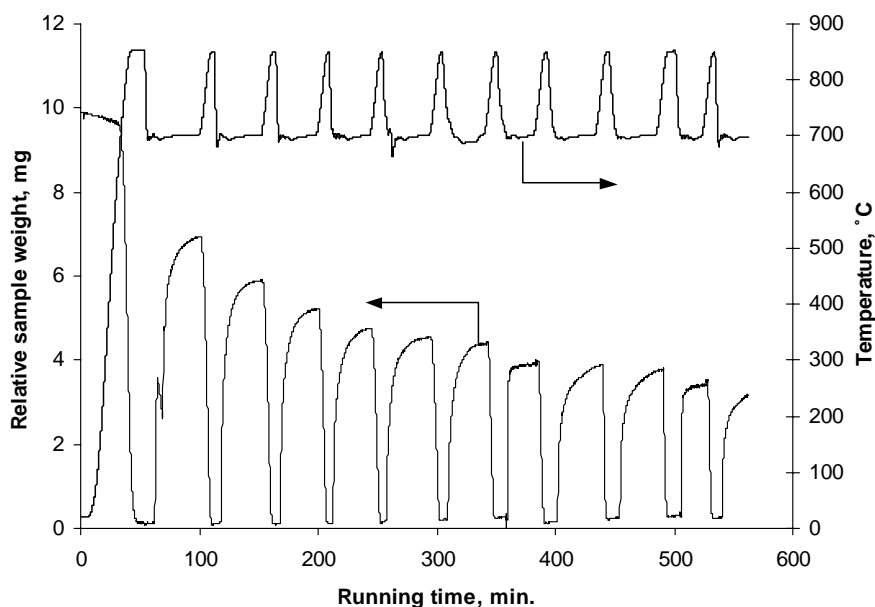


Fig. 4. TGA calcination/carbonation results for Cadomin limestone.

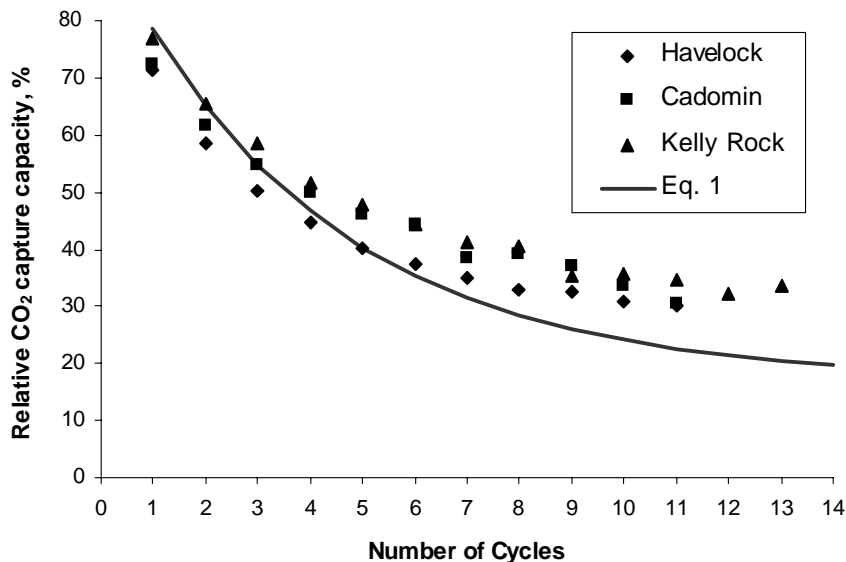


Fig. 5. Maximum carbonation capacity in TGA tests as a function of the number of cycles.

carbonation portion of the cycle exhibiting an initial rapid rate of mass increase followed by an abrupt transition to a slower rate of mass increase and eventual plateau. Similar observations were made for Havelock and Kelly Rock limestone. The total time required to complete 1 cycle is approximately 1 h.

Comparisons of CO₂ capture capacity of Cadomin, Havelock and Kelly Rock limestone over 13 cycles in the TGA are shown in Fig. 5. The solid line in this figure represents a semi-empirical model proposed for the decay of the capture capacity [7], based on TGA and fixed bed data and from other investigators for a number of limestone sorbents [9–13]:

$$X_N = f_m^N (1 - f_w) + f_w \quad (1)$$

where X_N is the maximum carbonation conversion achieved after N cycles, $f_m = 0.77$ and $f_w = 0.17$.

It is interesting to note that the TGA results of this study match the empirical model curve during the first 5 cycles. However, the TGA results show higher CO₂ capture capacity with increasing cycle number, leading to a notable difference after 10 cycles. In general, all three-limestone types follow the same trend, starting with a CO₂ capture capacity just under 80% and decaying to a final capacity of approximately 30% over 13 cycles. There are some slight differences in the capacity between specific limestone types, particularly during the first few cycles; however, these differences are negligible. This behavior is in contrast to results obtained from the FBC, where Havelock limestone showed a consistently higher capacity for CO₂ than Cadomin limestone.

The effects of calcination/carbonation cycling in the FBC are summarized in Fig. 6. For the sake of comparison, data from other workers are also presented in this figure. Once again the solid line represents the semi-empirical model curve (Eq. (1)). Nevertheless, there appears to be good agree-

ment between the Havelock results and the empirical curve. This is in contrast to the Cadomin data, which shows a consistently lower capacity for CO₂. Furthermore, unlike the Havelock results, the Cadomin data do not appear to level off, suggesting that limestone type may be a factor in apparent contradiction to the work of Abanades and Alvarez [7], which argues that limestone type is not a critical factor in determining maximum carbonation capacity. More work is needed on different limestone types to explain the previous discrepancies and understand how natural limestones perform in such cycles under relevant carbonation and calcination conditions.

With respect to the reactivation tests, the results of Na₂CO₃, NaCl and pure CO₂ reactivation strategies proved to differ significantly between the TGA and FBC work (see Fig. 7). Doping the limestone with Na₂CO₃ and NaCl decreased the overall CO₂ capture capacity dramatically in the FBC tests. For example, the CO₂ capture capacity of 0.5% NaCl-doped Havelock limestone, the most favorable of these results, dropped to 40% in the first cycle and 5% after 3 cycles, at which point test work was abandoned. During calcination the additives may have formed a coating over the surface of the lime grains [15]. This coating may have helped to block pores preventing CO₂ from being absorbed during carbonation.

This doping effect was observed for both Na₂CO₃ and NaCl in FBC tests but did not apparently occur in the TGA experiments. TGA data showed no effect for Na₂CO₃ doping but a drop in the initial CO₂ capture capacity to approximately 50% when limestone samples were doped with NaCl. This initial reduction was followed by an almost, slow decay to approximately 40% after 13 cycles. Given that the cumulative CO₂ capture capacity (over 13 cycles) was higher for doped vs. undoped limestone, the TGA data would suggest that the NaCl could be used to enhance CO₂ capture

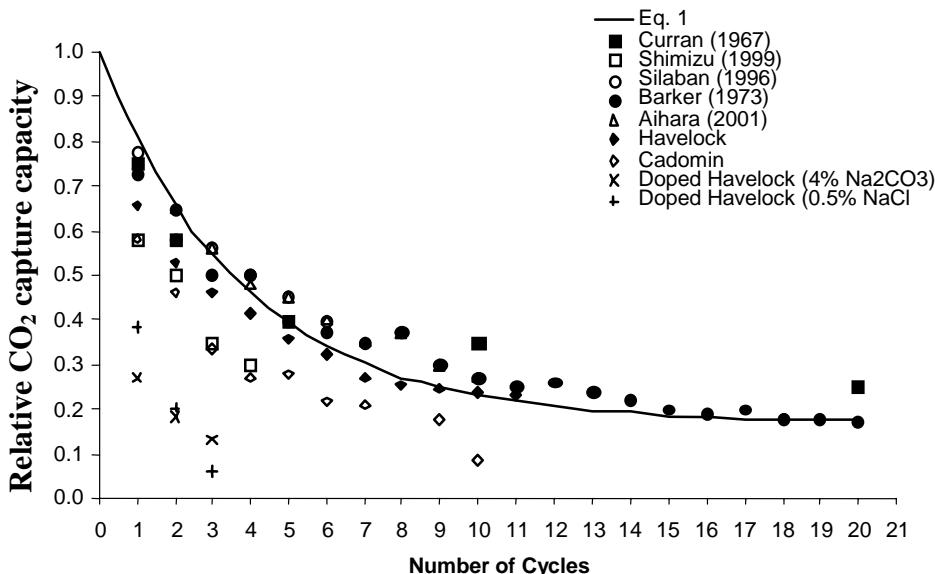


Fig. 6. Comparison of CO₂ capture capacities in the FBC environment for Havelock and Cadomin limestones.

capacity, since effective CO₂ removal demands that the limestone be used for many cycles, and the performance of modified sorbents during a few cycles is less important than the long-term behavior of the sorbent. However, for that to be the case it would be necessary to determine why TGA behavior is different from FBC performance.

A possible explanation for the different behavior of NaCl and Na₂CO₃ in the TGA environment may be provided from the evidence observed in the investigation on sulfation, where it appears that reactivation associated with NaCl is strongly dependent on the changes in pore size of the sorbent with increasing concentration of NaCl solution. It is possible to deactivate sorbents with too much NaCl ad-

dition [15,16], whereas the effect of Na₂CO₃ addition appears to be unrelated to the addition rate [16]. It has been speculated that Na₂CO₃ affects the sulfation rate primarily through its influence on the lattice structure by enhancing ion mobility [17,18]. In that case, it is easy to envisage how the NaCl might improve pore and surface area characteristics for optimum performance, whereas a mechanism involving improvement in ionic mobility might have no effect on enhancing CO₂ reaction performance.

Carbonating CaO in a pure CO₂ environment does not appear to be able to reactivate the sorbent based on the TGA results in agreement with results from Mess et al. [14]. Tests performed on Havelock limestone in the TGA showed no

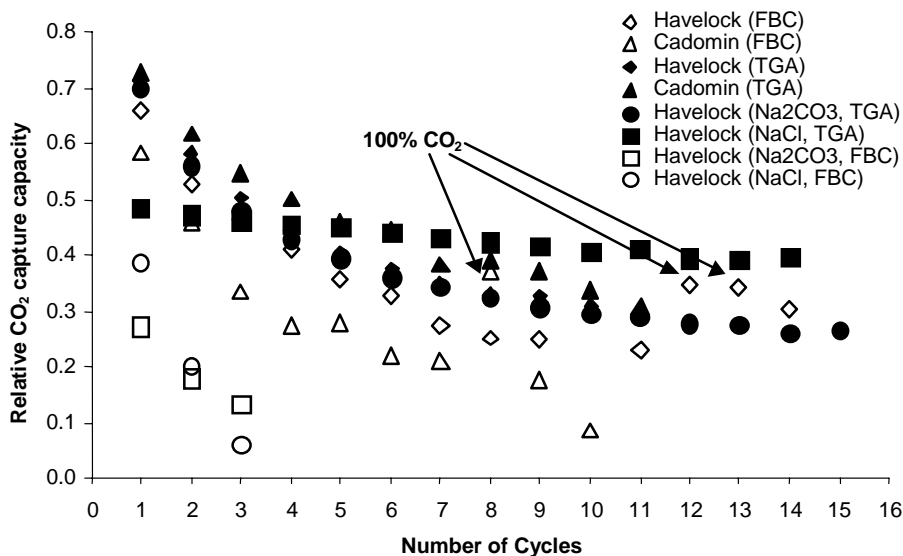


Fig. 7. Comparison of TGA and FBC reactivation strategies (open symbols refer to FBC data and closed symbols refer to TGA data).

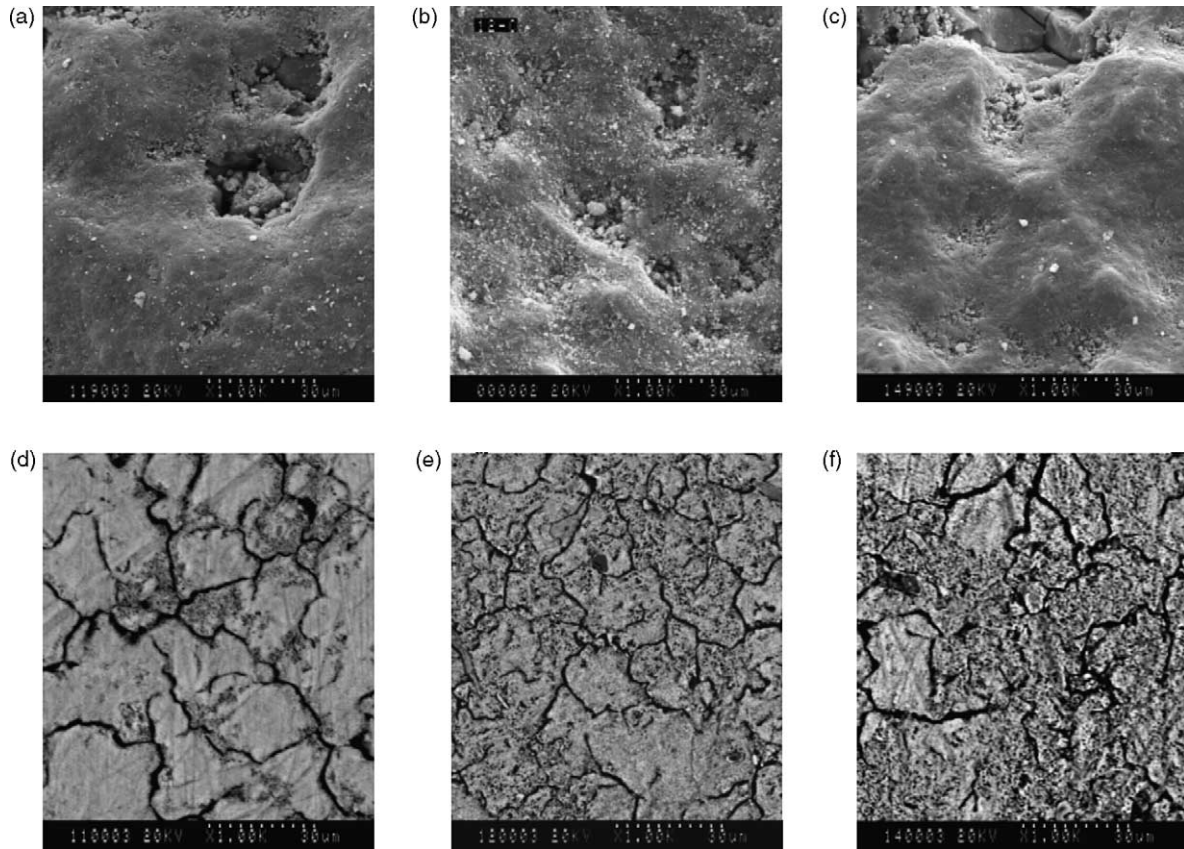


Fig. 8. SEM images—surface images of calcined samples: (a) cycle 11; (b) cycle 12; (c) cycle 14. Cross-section images of carbonated samples: (d) cycle 11; (e) cycle 12; (f) cycle 14 (cycles 11 and 14 were initially carbonated with 15% CO₂ in air, cycle 12 was initially carbonated with 100% CO₂).

appreciable increase in CO₂ capture capacity. FBC data, however, showed a marked rise in overall CO₂ capture capacity when either Havelock or Cadomin limestone was carbonated with pure CO₂. It was further noted that, when carbonation was carried out with 100% CO₂ for two successive cycles, Havelock limestone maintained a higher CO₂ capture capacity when next carbonated with 15% CO₂ in air. Although this is not a full reactivation option, this might be an interesting path to keep the activity of the carbonation/calcination cycle sufficiently high for a practical CO₂ capture process [5,6].

It was important to note that there was a significant improvement in CO₂ capture capacity when the lime was carbonated using pure CO₂ in FBC atmosphere, whereas the

impact was not observed in the TGA and other bench-scale work [9,14]. SEM photographs, however, did not show significant differences between samples carbonated with pure CO₂ or with 15% CO₂ and air. A comparison of surface photographs for calcined samples, which were originally carbonated at 15, 100 and 15% of CO₂, cycles 11, 12 and 14, respectively, are presented in Fig. 8. The same is true for images of the particle cross-sections. There is an apparent increase in pore size with increasing cycle number, but nothing that would distinguish the 100% carbonation sample from the 15% carbonation samples.

Interestingly enough, however, BET surface area measurements, pore volume and average pore sizes presented in Table 4 do indicate that carbonating with pure CO₂ does

Table 4
Surface area for Havelock limestone^a

Cycle	Pore volume (cm ³ /g)	Average pore size (Å)	BET (m ² /g)	Description
11	0.0045	200.8	1.24 ± 0.009	Carbonated sample collected at the end of a cycle
12	0.0015	116.9	0.60 ± 0.001	Carbonated sample collected at the end of a cycle
14	0.0052	219.3	1.17 ± 0.009	Carbonated sample collected at the end of a cycle
11	0.0121	319.7	2.89 ± 0.044	Calcined in oven at 900 °C
12	0.00306	387.6	1.07 ± 0.106	Calcined in oven at 900 °C
14	0.0918	286.9	2.48 ± 0.063	Calcined in oven at 900 °C

^a Cycles 11 and 14 (carbonation) were carried out in 15% CO₂ concentration, and cycle 12 was in pure CO₂.

influence the particle structure. The BET surface area, pore volume and average pore size for the two 15% samples, before and after carbonating in pure CO₂, are approximately the same, in spite of the fact they are separated by 3 cycles. The 100% carbonation sample consistently shows lower values for all these measured quantities.

Finally, particle size measurements (not shown here) were made after each cycle for the Havelock and Cadomin limestone tests in the FBC and indicated no appreciable attrition occurred. This rules out the possible explanation that the physical breakdown of particles could have resulted in the observed differences between the FBC and TGA experiments or between the Havelock and Cadomin limestone data from the FBC. A detailed investigation is in progress to elucidate on the discrepancies between TGA and FBC data reported above, in an attempt to bring to practice the encouraging reactivation results obtained in TGA and to enhance the partial reactivation observed in the FBC tests with pure CO₂.

4. Conclusions

Experiments were performed on three different limestone types—Havelock, Cadomin and Kelly Rock—in a TGA and an FBC. The objective of these experiments was to verify the effect of calcination/carbonation cycles on the capture capacity of CaO for CO₂. The TGA results showed relatively good agreement between the experimental data and the work of previous researchers, for the three limestones. FBC data also agreed well for Havelock limestone but there were significant discrepancies for the Cadomin limestone for which the overall CO₂ capture capacity decreased more rapidly and was significantly lower than the empirical curve. Thus, while these results are in general agreement with previous research, they suggest that the rate of decay of CO₂ capture capacity in CaO is not only a function of cycle number.

The present study also focused on several strategies for reactivating CaO using Na₂CO₃ and NaCl additives and carbonating the lime in pure CO₂. Na₂CO₃ and NaCl additives failed to reactivate the CaO having the reverse effect of severely reducing the CO₂ capture capacity in the FBC tests. TGA experiments on Havelock and Cadomin limestone showed no effect due to the addition of Na₂CO₃, but a marked improvement upon the addition of NaCl, raising the overall capacity to an almost constant value of 40% through 13 cycles. Carbonation of CaO in pure CO₂ showed differences between TGA and FBC results. TGA performance was

unaffected by carbonating in pure CO₂, irrespective of the limestone, whereas the FBC tests clearly showed an increase in CO₂ capture capacity for both Havelock and Cadomin limestones. These results suggest that comparing TGA and FBC experiments may not necessarily be simple, and indicate that caution is necessary when using TGA results in lieu of FBC data. The data presented here suggest that carbonating in pure CO₂ is able to partially reactivate CaO for CO₂ capture and should be examined further, along with NaCl doping.

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