

## Pilot testing of enhanced sorbents for calcium looping with cement production



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### HIGHLIGHTS

- HBr doping technique for improving calcium looping performance has been demonstrated at pilot scale.
- Attrition resistance of calcium sorbent at pilot scale has been demonstrated due to HBr doping.
- Successful production of cement from the residues has been demonstrated at the kg scale.

### ARTICLE INFO

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### ABSTRACT

One of the main challenges for commercialising calcium looping (CaL) as a CO<sub>2</sub> capture technology is maintaining a high level of sorbent reactivity during long-term cycling. In order to mitigate the decay in carrying capacity, research has moved towards producing enhanced sorbents. However, this creates potential problems related to ease of scaling up production techniques and production costs, and raises the question as to whether such approaches can be used at large scale. On the other hand, a key advantage of CaL over other carbon capture technologies is synergy with the cement industry, i.e., use of spent sorbent as a feedstock for clinker production. In this work two enhanced materials: (i) limestone doped with HBr through a particle surface impregnation technique; and (ii) pellets prepared from limestone and calcium aluminate cement, were tested in a 25 kW<sub>th</sub> dual fluidised bed pilot-scale reactor in order to investigate their capture performance and mechanical stability under realistic CaL conditions. Moreover, the spent sorbent was then used as a raw material to make cement, which was characterised for phase and chemical composition as well as compressive strength. The HBr-doped limestone showed better performance in terms of both mechanical strength and stability of the CO<sub>2</sub> uptake when compared to that of pellets. Furthermore, it was shown that the cement produced has similar characteristics and performance as those of commercial CEM 1 cement. This indicates the advantages of using the spent sorbent as feedstock for cement manufacture and shows the benefits of synthetic sorbents in CaL and suitability of end-use of spent sorbents for the cement industry, validating their synergy at pilot scale. Finally, this study demonstrates the possibility of using several practical techniques to improve the performance of CaL at the pilot scale, and more importantly demonstrates that commercial-grade cement can be made from the lime product from this technology.

### 1. Introduction

CO<sub>2</sub> emissions from the power generation and industrial sectors have increased rapidly in recent decades, and represent the greatest contributors to the greenhouse gas effect [1]. A portfolio of low-carbon technologies needs to be deployed in order to mitigate the effects of these emissions in many natural systems. Carbon capture and storage

(CCS) is part of this portfolio and has been proposed as a route for the decarbonisation of power generation and carbon-intensive industrial sectors [2,3].

Calcium looping (CaL) is a second-generation technology for CO<sub>2</sub> capture, which has attracted a fair amount of research activity [4–8]. Typically, a CaL system (Fig. 1) consists of two interconnected fluidised-bed reactors and is based on the reversible carbonation of lime. In the

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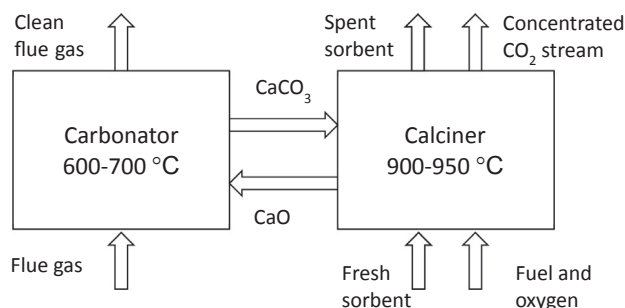


Fig. 1. Schematic of the calcium looping (CaL) cycle.

reactor, operating at  $\sim 650$  °C (carbonator), CaO-based material is carbonated with the  $\text{CO}_2$  present in flue gas or a gas stream from carbon-intensive industrial processes; the saturated sorbent is then transferred to a second reactor operating at  $\sim 900$  °C (calciner) where the sorbent is regenerated. In order to provide the heat for calcination, a fuel is burnt under oxy-fuel conditions in the calciner, while the high-grade heat generated in the carbonator is intended to be used in a steam cycle.

Due to sorbent deactivation, make-up of fresh and purge of deactivated material are required. One of the unique advantages that the CaL cycle has over other CCS technologies is its synergy with cement production. Namely, it is possible to decarbonise both the cement industry, which is responsible for 7–10% of global anthropogenic  $\text{CO}_2$  emissions [9], and the power sector by using the spent sorbent from  $\text{CO}_2$  capture as a feedstock for making clinker [6,10–12].

The CaL concept has been demonstrated at scales of up to 1.9  $\text{MW}_{\text{th}}$  with different reactor configurations. Some examples of these plants include: the 0.1  $\text{MW}_{\text{th}}$  plant in CanmetENERGY (Ottawa, Canada), the 0.2  $\text{MW}_{\text{th}}$  pilot plant in IFK (Stuttgart, Germany), the 1.7  $\text{MW}_{\text{th}}$  unit “La Pereda” run by CSIC in Oviedo, Spain, the 1.9  $\text{MW}_{\text{th}}$  unit in Taiwan, and the 1  $\text{MW}_{\text{th}}$  plant in Darmstadt, Germany [13–20].

However, one of the main challenges of this technology, namely the deactivation and loss of active material over the capture/regeneration cycles, still remains [21–23]. The loss of activity is generally attributed to sintering [24–26], attrition and fragmentation [27–29], ash deposition [30], and the competing sulphation reaction [31–33]. In order to overcome this issue, research has focused on the modification of natural materials and the development of synthetic sorbents with techniques such as sol-gel combustion [23,34–38], organic acid modifications [39–43], co-precipitation [44,45], granulation [46–52], and mineral-acid doping [53–55].

Al-Jeboori et al. [53,54] investigated doping of limestone with HBr, HCl,  $\text{HNO}_3$  and HI, and the effect on long-term cycling performance of doped sorbents. They demonstrated that such dopants can significantly improve long-term reactivity. The degree of improvement was found to be dependent on the type of limestone used, and the type and concentration of doping agent. The highest residual conversions were observed for Havelock and Longcal limestones doped with HBr (0.167 mol % HBr/ $\text{CaCO}_3$ ) [54]. Another effective technique for producing synthetic sorbents for fluidised bed utilisation is the production of calcium aluminate pellets that have been studied at different scales for reactivity and attrition behaviour [47,56]. It has been suggested that the better performance in these synthetic materials is linked to the formation of mayenite ( $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ ), which stabilises the sorbent morphology and mitigates sintering.

This work investigates the capture performance of enhanced CaO sorbents at the pilot scale (25  $\text{kW}_{\text{th}}$ ), under conditions anticipated for real industrial CaL systems. Natural limestone doped with HBr, employing a particle surface impregnation technique, and calcium aluminate pellets were tested in CaL cycles, and the suitability of spent material for use in the cement industry was then explored. The main goal of this study is the pilot demonstration of CaL with enhanced sorbents and its synergy with cement manufacture.

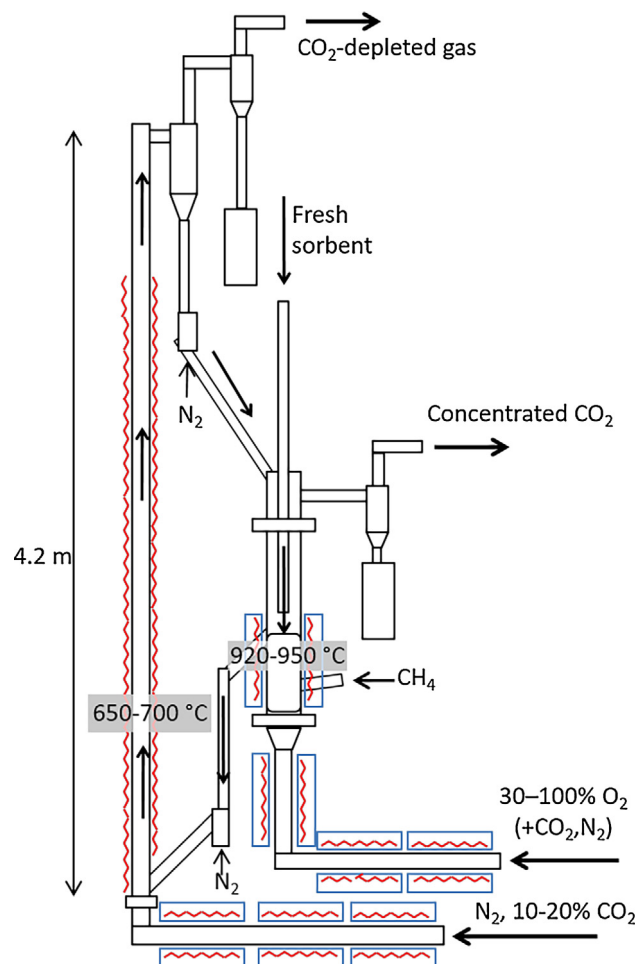


Fig. 2. Schematic of the 25  $\text{kW}_{\text{th}}$  pilot plant. Note: red lines denote heated sections.

## 2. Experimental

### 2.1. Materials

Longcal, a high-purity limestone provided by Longcliffe Ltd, UK, was used as a  $\text{CaCO}_3$  source. Commercial calcium aluminate cement, CA-14, manufactured by Almatiss, was used as a binder in the pelletisation process and as a source of  $\text{Al}_2\text{O}_3$ . The limestone-doping solution was prepared by diluting 57 g of 47%-wt. HBr with deionised water to form 1 L of 0.33 M solution.

### 2.2. Sorbent preparation procedure

Two types of enhanced sorbents were produced: (i) HBr-doped limestone (0.17 mol% HBr/ $\text{CaCO}_3$ ); and (ii) pellets containing 10 wt% calcium aluminate cement and 90 wt% calcined limestone. The pellets were prepared using a Glatt GmbH granulator. A comprehensive description of the pelletisation technique can be found elsewhere [47]. After pelletisation, the particles were sieved to the desired particle size range and air dried for 24 h. HBr was selected as a suitable dopant based on previous work [54]. However, the original doping technique was modified to enable a three-order-of-magnitude increase in production scale. Here, 20 kg of Longcal limestone was evenly sprayed with 1 L of 0.33 M HBr solution, and mixed thoroughly to obtain a uniform distribution of the dopant. The sample was then spread out and dried for several days under ambient conditions.

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