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From waste to high value utilization of spent bleaching clay in synthesizing high-performance calcium-based sorbent for CO_2 capture

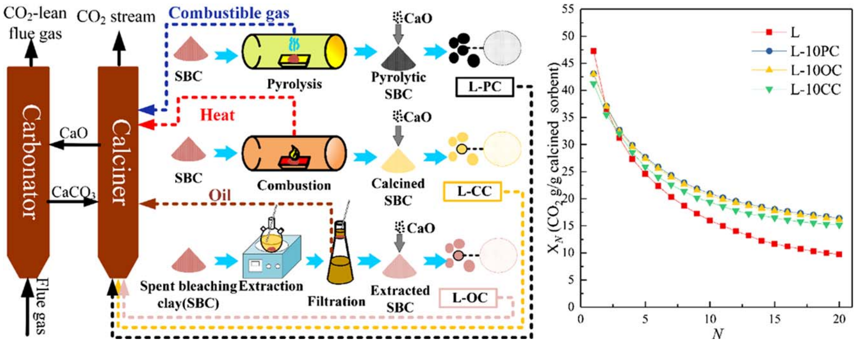
Chenglin Su[a], Lunbo Duan[a,*], Felix Donat[b], Edward John Anthony[c]

[a] *Key Laboratory of Energy Thermal Conversion and Control, Ministry of Education, School of Energy and Environment, Southeast University, Nanjing 210096, China*
[b] *Laboratory of Energy Science and Engineering, ETH Zurich, Leonhardstrasse 21, 8092 Zurich, Switzerland*
[c] *Centre for Combustion and CCS, School of Energy, Environment and Agrifood, Cranfield University, Cranfield, Bedfordshire MK43 0AL, UK*

HIGHLIGHTS

- A novel CaL process integrated with spent bleaching clay (SBC) recycle is proposed.
- New enhanced CaO-based sorbents were made from lime and regenerated SBC.
- The synthesized sorbents present good pore structures and enhanced cyclic performance.
- A synergistic effect is achieved for the sorbent doped with 5 wt.% pyrolytic SBC and 10 wt.% cement.

GRAPHICAL ABSTRACT



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ABSTRACT

A novel calcium looping (CaL) process integrated with a spent bleaching clay (SBC) treatment is proposed whereby fuels and/or heat from regeneration of SBC provide extra energy for the calcination process, in addition, the regenerated SBC can be used to synthesize enhanced CaO-based sorbents. Different kinds of composite samples were prepared with the regenerated SBC and/or aluminate cement at various doping ratios via a pelletization process. All pellets were subjected to thermogravimetic analyzer tests employing severe reaction conditions to determine the optimal doping ratios and regeneration method for the SBC based sorbents. These results demonstrate that pellets containing combustible components showed higher CO_2 uptakes, due to the improved pore structure, which was verified by N_2 adsorption measurements. The as-prepared sorbent "L-10PC" (90 wt.% CaO/10 wt.% pyrolytic SBC) achieved a final CO_2 uptake of 0.164 g(CO_2) g(calcined sorbent)$^{-1}$ after 20 cycles, which was 67.3% higher than that of natural limestone particle. A new larnite (Ca_2SiO_4) phase was detected by X-ray diffraction analysis, however the weak diffraction peak associated with it indicated a low content of larnite in the pellets, which produced a smaller effect on performance compared to cement. A synergistic effect was achieved for a sample designated as "L-5PC-10CA" (85 wt.% CaO/5 wt.% pyrolytic SBC /10 wt.% cement), which resulted in the highest final uptake of 0.208 g(CO_2) g(calcined sorbent)$^{-1}$ after 20 cycles. Considering the simplicity of pyrolysis regeneration process and the excellent capture capability of pellets doped by pyrolytic SBC, the proposed system integrating CaL with SBC pyrolysis treatment appears to offer particular promise for further development.

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[*] Corresponding author.
E-mail address: duanlunbo@seu.edu.cn (L. Duan).

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1. Introduction

With the Paris agreement, the reduction of anthropogenic CO_2 emissions is receiving increasing attention. To achieve the goals of that agreement, CO_2 must decline rapidly after 2030, with global CO_2 emissions from energy- and industry-related sources reaching net-zero levels between 2060 and 2080 [1]. Post-combustion carbon capture and storage (PCCS) remains an essential part of such a strategy, incorporating existing energy-intensive industries, such as coal-fired power plants, cement and iron-and-steel production, which constitute the top three anthropogenic sources of CO_2 emission [2–4].

Calcium looping (CaL), as one of the newer and promising method of PCCS, has been studied extensively and appears to be an important rapidly developing PCCS technology [5–9]. Limestone is a cheap and abundant natural material, making it particularly suitable as sorbent for the CaL process [6,9–11]. However, although 1.7 MW_{th} [7] and 1.9 MW_{th} [8] demonstration projects have been successfully tested, the cyclic performance degradation and attrition loss of sorbent are still two major obstacles that need to be overcome. The CO_2 capture capacity decreases rapidly (about 15% decay per cycle for the first five or six cycles) due to thermal sintering related to the low Tammann temperature of $CaCO_3$ (533 °C) [12,13]. It was shown that the cyclic performance of lime-based sorbents can be improved substantially by the presence of a stable phase (with high $T_{Tammann}$). Several kinds of inert materials, including Al_2O_3, [14–19] MgO, [17,19–22] SiO_2, [13,23–25] ZrO_2, [17,19,26] and Y_2O_3 [19,27] have been shown to effectively improve the cyclic stability of the sorbent. It also appears possible that the spent CaO sorbents from CaL process can be reused to produce cement, which offers potential for a decrease by at least half of the CO_2 emissions from that industry, owing to the energy-intensive calcination procedure required when using natural limestone [3,28]. In view of the requirement of a typical clinker product, which consists of 67% CaO, 22% SiO_2, 5% Al_2O_3, 3% Fe_2O_3 and 3% others [13], components like Al_2O_3 and/or SiO_2 supported lime-based sorbents are likely to be more suitable for subsequent cement production.

Although CaL has been shown to be more cost-effective than other CO_2 capture methods [29], its cost could be dramatically reduced if such sorbents were prepared partially or totally from wastes. Li et al. [16] prepared carbide slag-derived $Ca_3Al_2O_6$-stabilized sorbents, which exhibited favorable capture performance compared to the original carbide slag itself. $Ca_3Al_2O_6$ has also been reported as an inert material in other work [17,18]. Yan et al. [30] reported a green synthesis of thermally stable CaO-based sorbents by adding fly ash, and the results of measurements demonstrated that $Ca_2Al_2SiO_7$ could help sorbents resist sintering. In particular, rice husk ash (containing 93 wt.% SiO_2) was identified by Li et al. [31] and Chen et al. [32] as particularly promising in enhancing the cyclic capture performance of synthetic sorbents. Yan et al. [33] first introduced nanosilica derived from photovoltaic waste ($SiCl_4$) into synthetic lime-based sorbents. The capture performance tests revealed that such sorbents exhibited pronounced improvement in performance, even under severe calcination conditions (e.g. at 920 °C in pure CO_2), in which larnite (Ca_2SiO_4) played a role for the stabilization. Tian et al. [34] successfully proposed an efficient CO_2 capture process wherein Ca and Fe were recovered from steel slag to prepare high-purity CaO-based sorbents for CO_2 capture and to obtain high-quality iron ores for steel production. It is therefore clear that the use of wastes in the PCCS process is an important option.

Because of its high specific surface, activated bleaching clay is widely used in the process of petroleum refining and edible oils refinery to remove the coloring and unstable impurities. It has been estimated that around 2 million metric tons of spent bleaching clay (SBC) are produced yearly worldwide based on 1 wt.% bleaching earth usage for about 200 million metric tons of edible oils and fats refined globally [35]. Usually, SBC contains ~20–40 wt.% residual oils. The disposal of SBC causes environmental pollution due to the presence of the waste oils and often requires expensive remediation of such sites. Currently, several methods have been proposed to recycle these oils and to ensure that SBC disposal is environmentally benign, and these approaches include the thermal treatment [36–39], solvent extraction [38,40–42], involving organic solvents [38,41,42], acid solutions [39,43], hot alkali solutions [37,44] and inorganic salt solutions [43,45]. Among these, organic solvent extraction has been repeatedly reported to be an appropriate method for SBC treatment. Wang et al. [42] proposed a process whereby step-by-step solvent extraction was used to recover the adsorbed oil from the lubricating oil refining clay: first, the spent clay is extracted by petroleum ether; second, this is followed by extraction with a mixture of petroleum ether and ethanol (95 vol%) two or three times; and then the resulting material is extracted by ethanol (95 vol%), followed by water washing; finally, the clay is dried at 130 °C to obtain a regenerated clay. Although > 99 wt.% of the adsorbed oil can be recovered by these procedures, such sophisticated processes are so complicated that they are economically infeasible. Thermal treatment, namely calcination in air, is usually regarded as a substandard, albeit cheap method to recover SBC, as it leads to the destruction of the clay's structure and the release of pollutants [42].

The studies on SBC regeneration are mainly aimed at recovering oil and achieving its reuse [37–39]. It should be noted that the fresh bleaching clays usually come from bentonite activated by acid treatment, the main components of which are SiO_2 and Al_2O_3, so as regenerated SBC. As noted above, SiO_2 and Al_2O_3 are good inert phases to mitigate the sintering of lime-based sorbents. As a result, the regenerated SBC can potentially be used for preparing CO_2 capture sorbents with lime. In general, any CaL CO_2 capture process contains two coupled reactors, namely the carbonation and calcination reactor, respectively. In the calcination reactor, it is necessary to consume extra fuels to achieve and maintain a high reaction temperature of 900 °C. Here, the fuels and heat can serve to provide extra energy consumed in calcination reactor as part of the regeneration process for the SBC. Furthermore, the residual flammable components in treated SBC will increase the porosity of any synthetic sorbents.

Currently, there is no research on the incorporation of SBC treatment into the CaL process and hence we propose a potential CO_2 capture system (shown in Fig. 1) whereby fuels (oils and combustible gases) and/or heat from different SBC regeneration steps enter the calciner. Three different treatment methods of the SBC are suggested including combustion, pyrolysis and organic solvent extraction. The solid residues are separately introduced into a pelletizer to synthesize sorbent pellets with calcined lime powder. Here, the fresh sorbents are then introduced into calciner, meanwhile the spent materials purged from the calciner are sent to the rotary kiln for cement production. A small amount of the cement can also be sent back to the pelletizer as dopant. In order to further examine the synthesis methods, different types of sorbents were prepared with various regenerated SBC by pelletization in this paper and CO_2 capture tests were then carried out to determine the optimal doping ratios and regeneration method of SBC for this system.

2. Experimental section

2.1. Materials

Fresh bleaching clay (FBC) and spent bleaching clay (SBC) studied in this paper were obtained from Sinopec Corp. China, and the SBC comes from lubricating oils refining process. Prior to examination by X-ray fluorescence, the spent clay was dried overnight in a hot air blast drying oven at 110 °C and its main components were shown to be SiO_2 and Al_2O_3 (Table S1, Supplementary Material). As shown in Table S2, there are a small amount of impurities such as sulphur, nitrogen and metals in SBC, but these values are far lower than that in coal. Natural limestone containing ~98.6 wt.% $CaCO_3$ was obtained from Omya Fine Chemical Industry Co., Ltd., Nanjing, China.

The oil content in SBC was determined as follows: Firstly, both the

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