

Study of CO₂ cyclic absorption stability of CaO-based sorbents derived from lime mud purified by sucrose method

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Abstract Using lime mud (LM) purified by sucrose method, derived from paper-making industry, as calcium precursor, and using mineral rejects–bauxite-tailings (BTs) from aluminum production as dopant, the CaO-based sorbents for high-temperature CO₂ capture were prepared. Effects of BTs content, precalcining time, and temperature on CO₂ cyclic absorption stability were illustrated. The cyclic carbonation behavior was investigated in a thermogravimetric analyzer (TGA). Phase composition and morphologies were analyzed by XRD and SEM. The results reflected that the as-synthesized CaO-based sorbent doped with 10 wt% BTs showed a superior CO₂ cyclic absorption–desorption conversion during multiple cycles, with conversion being >38 % after 50 cycles. Occurrence of Ca₁₂Al₁₄O₃₃ phase during precalcination was probably responsible for the excellent CO₂ cyclic stability.

Keywords Lime mud · Sucrose method · Cyclic capture CO₂ · Rejects–bauxite-tailings · Precalcination · Doping

Introduction

Anthropogenic CO₂ emission is the primary reason for global warming (Kargari and Mastouri 2011). It is well accepted that CaO is a kind of sorbents most likely to be industrialized (Li et al. 2013; Liu et al. 2012) due to its affordability and high CO₂ adsorption capacity. Calcium looping cycle reaction between

CaO and CaCO₃ is reversible and always operates at high temperatures (650–800 °C) (Luo et al. 2013). Operating at high temperatures is advantageous for heat recovery and reasonable use of the additional heat generated from CaCO₃ decomposition. But multi-cycles of carbonation–calcination under high temperatures cause the rapid decaying of the conversion of CaO-based sorbents (Chen et al. 2013; Liu et al. 2012).

As for the reduction of conversion within multi-cycles, different preparation methods are developed to improve it (Reddy et al. 2014; Sanchez-Jimenez et al. 2014; Wang et al. 2013; Zhang et al. 2014b; Zhenissova et al. 2014). The most common method of enhancing CO₂ cyclic absorption–desorption conversion is to embed CaO into a thermostable skeleton (Reddy et al. 2014; Yu and Chen 2014). Alumina is a common dopant to provide stable structure. Broda et al. (2014) used aluminum nitrate as aluminum precursor to prepare the CaO-based sorbent which had a homogeneous morphology and stable conversion of 0.56 g(CO₂)/g(sorbent)⁻¹ in the 30th cycle. Angeli et al. (2014) also used aluminum nitrate and triethanolamine as complex agent to prepare the sintering-resistant CaO-based sorbent. Triethanolamine was used to make the Al³⁺ and Ca²⁺ disperse homogeneously in the sorbent skeleton, and the mixture was calcined to form interparticle voids which could decelerate sintering. Chen et al. (2013) synthesized CaO-based sorbent by using aluminate cement as dopant. The high mechanical strength of aluminate cement can improve the mechanical property of CaO-based sorbent as well.

In consideration of the energy demand for CO₂ cyclic absorption–desorption process (Liu et al. 2012; MacKenzie et al. 2007), how to reduce the sorbent cost in a sensible way needs to be further studied. Solid wastes, occupying land and causing potential harm to environment and human health, have become one of the major adverse factors for industry and urban development (Zhang et al. 2014a). Mineral rejects–bauxite-tailings (BTs), generated from the aluminum

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production of the Bayer process, were used as alumina source in our previous study (Hu et al. 2015). The analytical pure CaCO₃ powder doped with 5 % BTs obtained a superior CO₂ absorption conversion of 42.57 % after 50 carbonation–calcination cycles.

Some solid wastes containing CaO, such as steel slag (Zhang and Hong 2011; Salman et al. 2014), eggshell (Witoon 2011), and carbide slag (Li et al. 2015), have been used as calcium precursors to absorb CO₂. Lime mud (Li et al. 2012; Sun et al. 2013; Zhang et al. 2014a), a kind of toxic industrial waste, comes from the causticization reaction in alkali recovery process of paper-making industry and its major chemical component is CaCO₃. China has occupied approximately 50 wt% of the global paper yield (over 50 million tons per year). One ton of pulp produces about 0.5 t of lime mud (LM) (He and Barr 2004). Therefore, it is urgent to find out one way to reuse LM. So far, LM has been used in waste water treatment (Zhang et al. 2011; Zhang et al. 2013) and gas sorbents (Li et al. 2012; Sun et al. 2013). Li et al. found out that LM had higher sulfation/carbonation capacity than limestone (Li et al. 2012; Sun et al. 2013). However, they washed LM with distilled water to remove soluble impurities, which probably generated a great deal of waste water. Sucrose method (Dai et al. 2015) is generally used for measuring the content of active calcium in limestone to extract active calcium oxide from LM. Through this method, some insoluble impurities were separated by filtration, and CaCO₃ formed by treating calcium sucrose solution with CO₂ for separating soluble impurities (Dai et al. 2015). In addition, sucrose solution can be reused to extract active calcium oxide from the LM of paper-making industry.

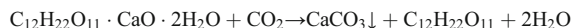
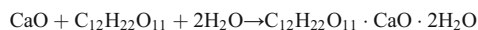
As far as I am concerned, there are no reports on using the Ca wastes purified by sucrose method to adsorb CO₂. In this study, using LM purified by sucrose method and BTs as calcium source and dopant, the CaO-based sorbents were developed. Influences of BTs content, calcining temperature, and time on cyclic absorption properties were illustrated. In the meanwhile, the 15 % CO₂ carbonation atmosphere was used to simulate power plants tail gas in a thermogravimetric analyzer (TGA).

Experimental

Materials and sample preparation

LM (Yunnan Yunjing Forestry & Pulp Co., Ltd) was precalcined at 760 °C for 4 h in the muffle furnace to obtain calcium oxide. The precalcined LM was mixed with 0.4 mol/L sucrose solution and strongly agitated for 15 min. Then the residues were separated by

filtration. The filtrate was slowly treated with CO₂ to form CaCO₃ (CBLM). Washed CBLM with distilled water and dried until constant weight. The reactions were as follows:



According to the above reaction equations, sucrose solutions and CO₂ can be recycled by filtration and calcining the CaCO₃, respectively. The detailed process is shown in Fig. 1.

BTs powder (425 mm, Zhongzhou Branch China Aluminum Co., Ltd.) was used as dopant. The main composition of LM and BTs is shown in Table 1. CBLM and BTs (5–20 wt%) were weighed, then distilled water was added to get a suspension. The suspension was stirred for 15 min and then ultrasonically dispersed 20 min to obtain a well-mixed precursor. Drying and calcining the precursor at 800 or 900 °C for 3–5 h in air, CaO-based sorbents were fabricated (in the paper, CBLM-5-800-4 means the CBLM doped with 5 wt% BTs and the mixture was precalcined 4 h at 800 °C; CBLM-10 means the CBLM doped with 10 wt% BTs without precalcining).

Adsorbent testing

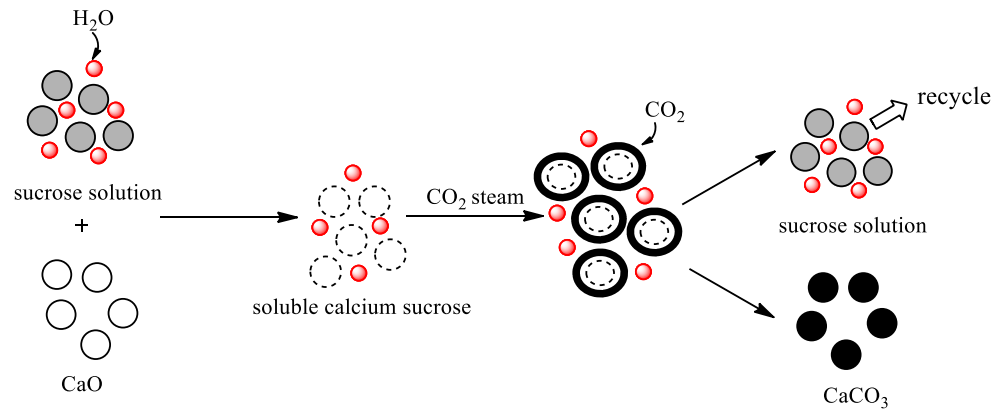
The cyclic carbonation–calcination testing was carried out in the thermogravimetric analyzer (STA 449 F3, Netzsch Co. Ltd., Germany). The temperature of TGA was heated to 750 °C at a rate of 20 °C/min under a N₂ gas flow of 50 mL/min. Once 750 °C was reached, a CO₂ flow of 50 mL/min was also introduced into TGA and this condition would be kept for 10 min for CO₂ absorption. The calcination was performed under a N₂ gas flow of 100 mL/min for 5 min. The conversions of sorbents were calculated as follows:

$$X = \frac{m - m_0}{\xi m_0 \varphi}$$

X is the carbonation conversion of CaO-based sorbents, m_0 is the sample mass after calcination, m is the sample mass during carbonation, φ is the CaO content of CaO-based sorbents, and ξ is the mole mass ratio of CaO with CO₂.

The phase compositions of the as-prepared sorbents were identified by XRD (D/MAX-RA, Rigaku, Japan) at room temperature using a Cu K_α radiation. The morphologies of CaO-based sorbents were performed in a FEI Quanta 200 apparatus (JSM-35C, JEOL Ltd., Japan).

Fig. 1 The process of the preparation of CaCO₃ (CBLM)



Results and discussion

Phase composition of CaO-based sorbents

Figure 2a shows the XRD pattern of LM and CBLM. CBLM has stronger peaks of CaCO₃ than LM. XRD pattern of BTs is shown in Fig. 2b. As reported in our previous study, BTs were composed of Al₂O₃, Fe₉TiO₁₅, SiO₂, Fe₃O₄, TiO₂ and Ca_{0.986}(Ti_{0.605}Al_{0.349}Fe_{0.023})Si(O_{0.508}(OH)_{0.492})O₄ phases. No CaO phase was detected. For CBLM doped with BTs, dicalcium silicate (Ca₂SiO₄), gehlenite (Ca₂Al₂SiO₇), kalsilite (KAlSiO₄), and mayenite (Ca₁₂Al₁₄O₃₃) phases can be detected according to Fig. 2c. It is noticed that precalcining temperature has obvious influence on phase composition. The peak intensity of Ca₂SiO₄ and Ca₂Al₂SiO₇ were stronger at higher precalcining temperature, which showed that higher temperature accelerated the reactions between LM and BTs. Inert material Ca₁₂Al₁₄O₃₃ formed by the reaction between Al₂O₃ with CaO (Li et al. 2015; Yu and Chen 2014). It was reported that Ca₁₂Al₁₄O₃₃ (Hu et al. 2015; Zhou et al. 2012) can enhance the cyclic durability of sorbents without participating in the capture and release of CO₂. However, it is uncertain whether Ca₂SiO₄ (Li et al. 2009; Wang et al. 2008), Ca₂Al₂SiO₇, and KAlSiO₄ play a positive role during cyclic adsorption/desorption. Wang et al. (2008) found out that Ca₂SiO₄ sorption decayed from 45.68 to 20.79 % after undergoing 13 cycles in a mixture of 15 vol% CO₂ with 85 vol% N₂ atmosphere. The main reason for decay of Ca₂SiO₄ lies in the decomposition of Si–O chain, powder growth, and sintering. While Manovic and Anthony (2009) reported that the existence of Ca₂SiO₄ is disadvantageous for CO₂ cyclic capture property.

Effect of different BTs content on CO₂ cyclic absorption property

Figure 3 shows cyclic conversion of CBLM doped with 5–20 wt% BTs. This figure reflected that the CBLM-5-800-4 exhibited a relatively high conversion in the first four cycles. However, CBLM-10-800-4 (from 45.7 to 38.8 %) had more stable conversions than CBLM-5-800-4 (from 42.5 to 28.5 %) after the 6th cycle. As shown in Fig. 3, an appropriate amount of dopant is in favor of improving cyclic absorption stability. On the other hand, dopant dispersed homogeneously in the crystal skeleton may provide a better contact between CaO and CO₂ (Hu et al. 2015; Wang et al. 2008). CBLM-20-800-4 shows a stable cyclic capacity but a lower conversion (from 33.7 to 22.8 %), mainly due to the large consumption of active calcium oxide to form inert materials (see XRD patterns).

Figure 4 compares the cyclic conversion of LM, CBLM, LM-10-800-4, and CBLM-10-800-4 under the same conditions. As shown in Fig. 4, the conversion of LM declined sharply (from 32.6 to 6.3 %), which was similar to the research results of Li et al. (2013). To improve the conversion of LM, Li et al. (2013) pre-washed LM with distilled water and found out that until the Cl⁻/Ca molar ratio went below 0.25:100, there was no negative effect on absorption conversion. But this process probably generated a great amount of waste water containing Cl⁻, which caused a huge burden to the environment. In this paper, it was obvious that the LM purified by sucrose method had the better cyclic adsorption durability. CBLM-10-800-4 possessed much

Table 1 Chemical composition of lime mud and BTs (wt%)

| Sample | CaO | CO ₂ | SiO ₂ | T _{Fe} | TiO ₂ | Al ₂ O ₃ | SO ₃ | Na ₂ O | K ₂ O | Other |
|----------|-------|-----------------|------------------|-----------------|------------------|--------------------------------|-----------------|-------------------|------------------|-------|
| Lime mud | 52.61 | 43.07 | 0.85 | 0.52 | 0.04 | 0.38 | 0.57 | 1.20 | 0.16 | 0.6 |
| BTs | 0.72 | | 25.48 | 7.53 | 2.97 | 37.12 | | | | 26.28 |

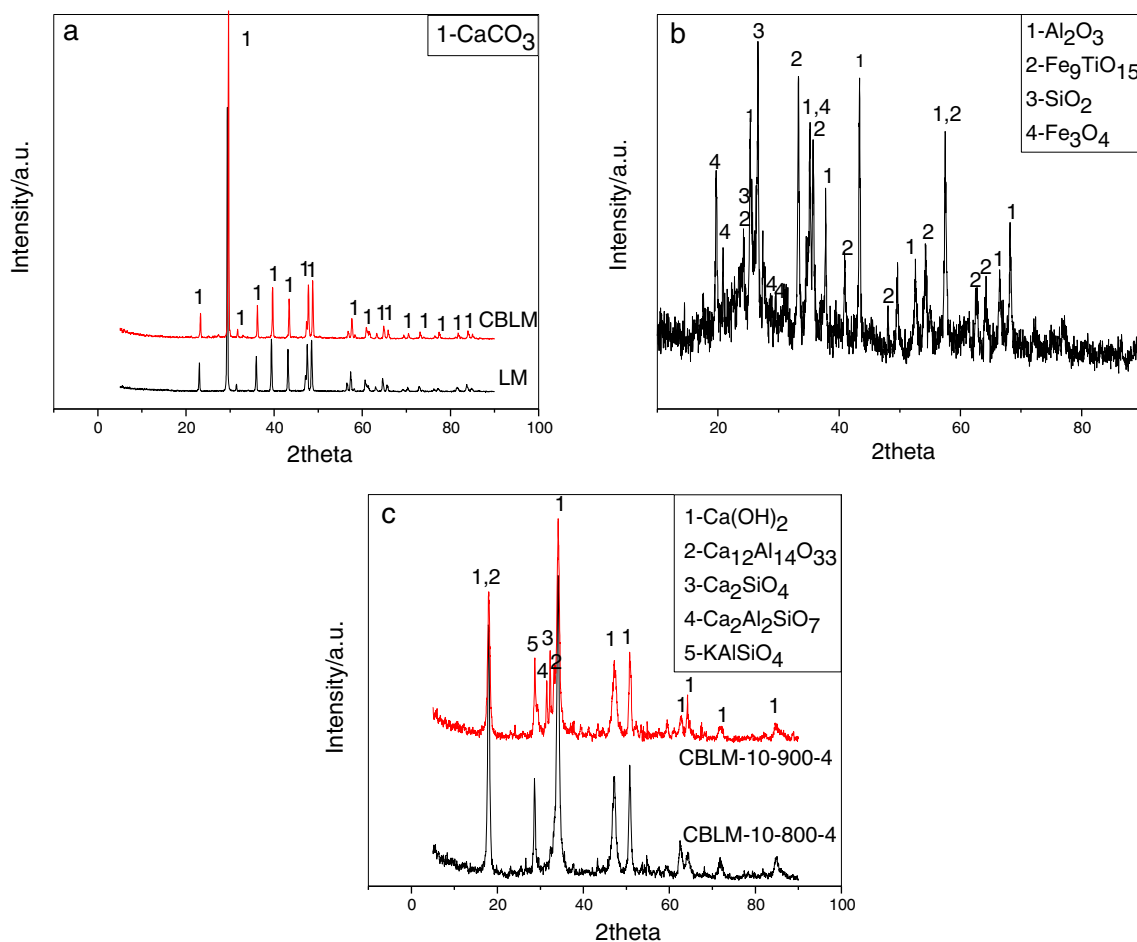


Fig. 2 a The XRD pattern of LM and CBLM. b BTs. c The comparison of CBLM-10-800-4 and CBLM-10-900-4

higher absorption conversion than LM-10-800-4 (from 36 to 21 %), which confirmed that the impurities from LM had negative effects on cyclic conversion. After

purification and doping BTs, there was a 32.5 % improvement of conversion at the 15th cycle (LM 6.3 %, CBLM-10-800-4 38.8 %).

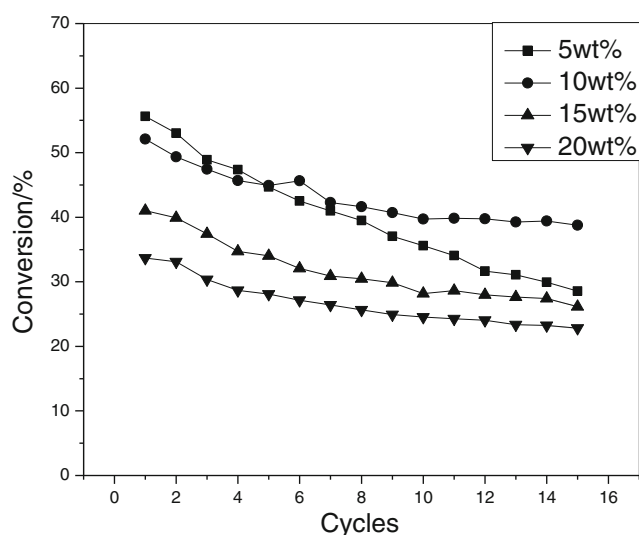


Fig. 3 Conversion of CaO-based sorbents doped with different BTs content in TGA (carbonation: 750 °C, 10 min, 50 % CO₂/50 % N₂; calcination: 750 °C, 5 min, 100 % N₂)

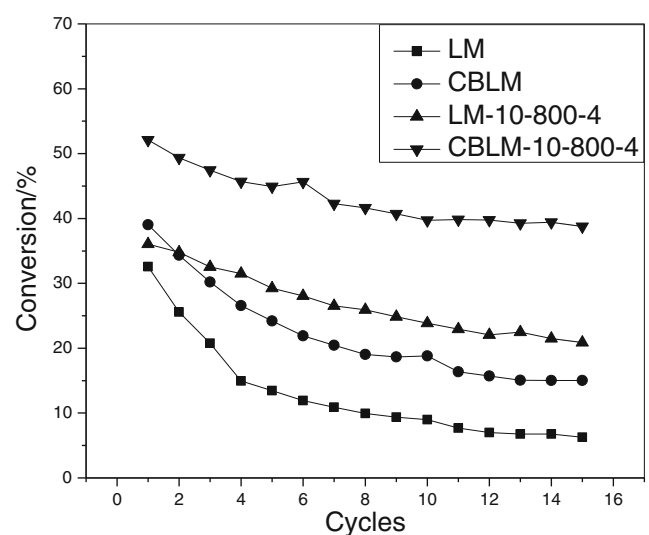


Fig. 4 The cyclic conversion comparison of LM, CBLM, LM-10-800-4, and CBLM-10-800-4 in TGA (carbonation: 750 °C, 10 min, 50 % CO₂/50 % N₂; calcination: 750 °C, 5 min, 100 % N₂)

Effect of different precalcining time and temperature on CO₂ cyclic absorption property

Figure 5 shows the cyclic absorption–desorption conversion of CBLM pretreated for 3–5 h at 800 °C. It can be seen from Fig. 5 that a superior cyclic conversion was obtained with a precalcining time of 4 h, which indicated that the appropriate precalcining time is beneficial for ion diffusing and rebuilding a more stable structure (Borgwardt 1989). The cyclic adsorption durability decreased with increasing precalcining time (5 h), which is because longer time caused the sintering of the sorbents (Butler et al. 2014; Borgwardt 1989).

Figure 6 shows the conversions of CBLM-10-800-4, CBLM-10-900-4, and CBLM-10 (without precalcination). Compared with CBLM-10-900-4 and CBLM-10, CBLM-10-800-4 had the better cyclic adsorption properties. The worse cyclic adsorption property for CBLM-10 is probably due to the stable skeleton structure has not formed during the initial cycles, which resulted in decaying rapidly of the conversions from the 1st cycle to the 3rd cycle. The lower cyclic adsorption property for CBLM-10-900-4 probably resulted from the lower surface areas and pore volumes. It is reported that surface areas and pore volumes had a direct influence on the final conversion, because the former affected the rate of carbonation and the latter affected the entry of gas into the internal surface of sorbents for absorption (Butler et al. 2014). In addition, Borgwardt (1989) found out that the sintering rate of limestone at 900 °C was one order of magnitude higher than that at 800 °C in inert atmosphere. Therefore, the higher the precalcining temperature was, the faster the surface areas and pore volumes declined (Fig. 8(e, f)).

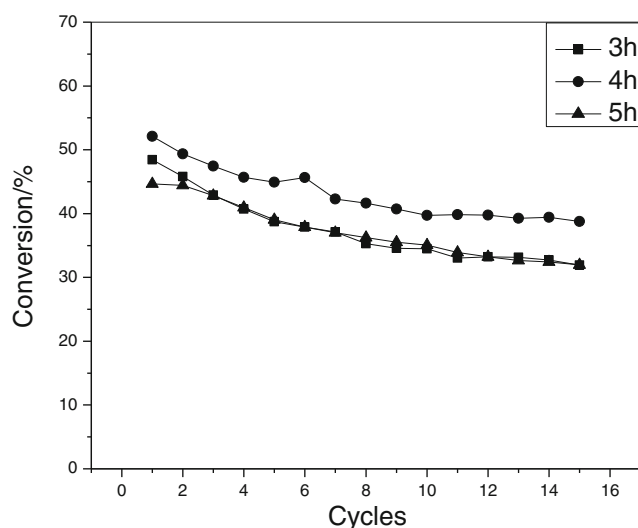


Fig. 5 Effects of precalcining time on carbonation conversion of CaO-based sorbents in TGA (carbonation: 750 °C, 10 min, 50 % CO₂/50 % N₂; calcination: 750 °C, 5 min, 100 % N₂)

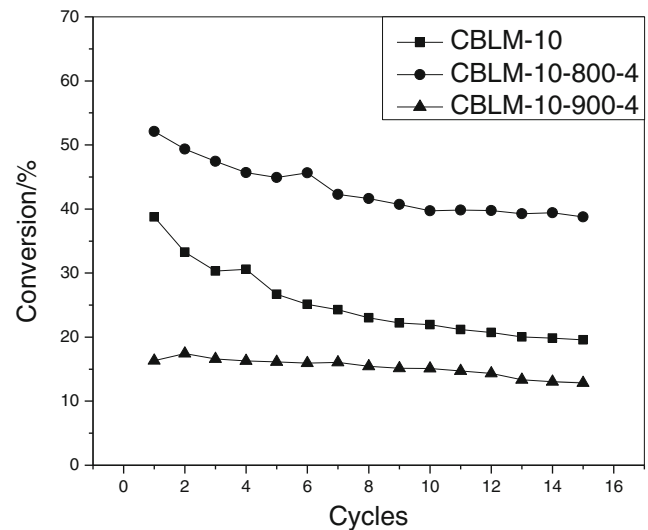


Fig. 6 Effects of precalcining temperature on carbonation conversion of CaO-based sorbents in TGA (carbonation: 750 °C, 10 min, 50 % CO₂/50 % N₂; calcination: 750 °C, 5 min, 100 % N₂)

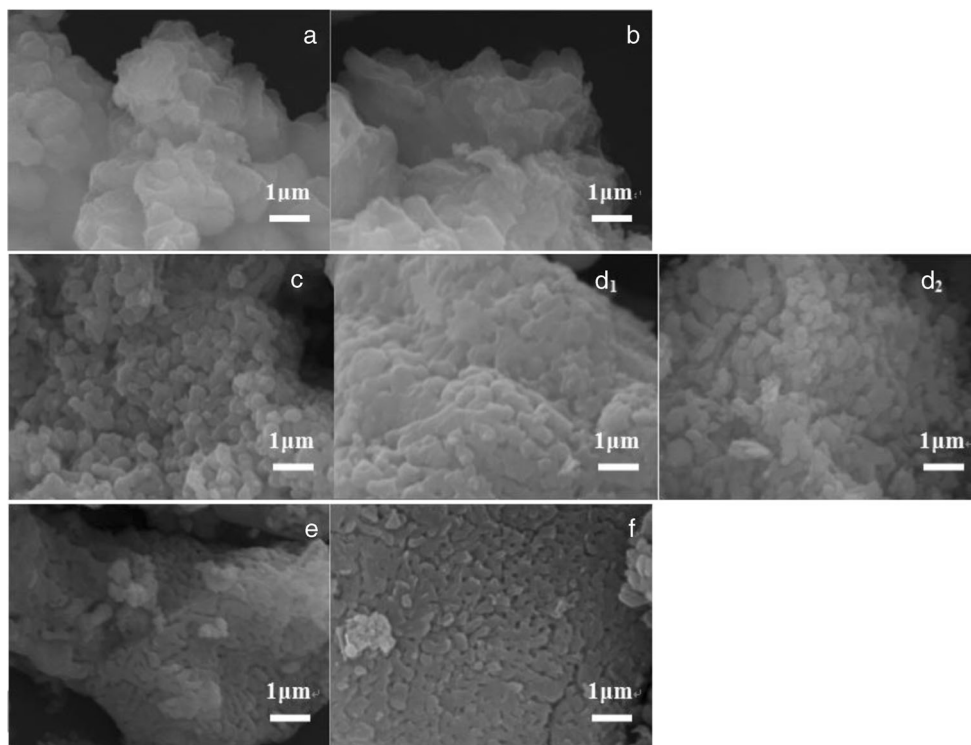
Adsorption property of a long series cycles with carbonation at 15 % CO₂ atmosphere

High driving force can enhance the initial carbonation rate with an increasing partial pressure of CO₂ (Yu and Fan 2011). To obtain the best performance of sorbent, the 50 % CO₂ was selected for carbonation. The existing coal-fired power plants are generating electricity by burning fossil fuels, with the exhaust volume fraction of CO₂ standing around 15 % in most cases (Liu et al. 2012). Therefore, CBLM-10-800-4h was treated with 15 % CO₂ atmosphere in order to simulate power plants tail gas. Figure 7 reflects the cyclic conversion of CBLM-10-800-4 carbonation at 15 % CO₂ atmosphere during 40 cycles, with the conversion ranging from 42.4 to 32.3 %. Compared with the CaO-based sorbents derived from the modified steel slag reported by Tian et al. (2015), the resultant CaO-based sorbents had better cyclic stability.

Morphologies of CaO-based sorbents

Figure 8 shows the morphologies of LM, CBLM, and doped CBLM (CBLM-10-800-4 and CBLM-10-900-4). It could be concluded from the three figures (Fig. 8(c, d₁, and d₂)) that the particles aggregated to some extent with multiple cycles and higher CO₂ concentration is apt to produce sintering. For CBLM-10-900-4, no obvious morphology differences were observed before and after cycle (Fig. 8(e, f)), mainly due to the rapidly coarsening of pores with high-temperature precalcination.

Fig. 8 Morphologies of LM (*a* after precalcination), CBLM (*b* after precalcination), CBLM-10-800-4 (*c* after precalcination, *d*₁ after the 15th calcination with 50 %CO₂ carbonation atmosphere, *d*₂ after the 40th calcination with 15 % CO₂ carbonation atmosphere), and CBLM-10-900-4 (*e* after precalcination, *f* after the 15th calcination)



Conclusions

Using lime mud (LM) purified by sucrose method and bauxite-tailings (BTs) as raw materials, the CaO-based sorbents for high-temperature CO₂ capture were fabricated and tested for multiple CO₂ capture–release cycles. Effects of precalcination conditions, dopant contents, and carbonation atmospheres on the cyclic adsorption properties of CO₂ were investigated. This research results showed that CBLM-10-800-4 exhibited a better cyclic conversion during 40

capture–release cycles (from 45.7 to 38.8 %). In comparison with LM, the conversion of CBLM-10-800-4 with the appropriate precalcination conditions and doping content improved by 32.5 % (after 15 cycles). Higher CO₂ partial pressure may improve the carbonation rate and conversion at the same time. But lower CO₂ partial pressure can obtain a more stable cyclic adsorption conversion during 40 cycles (from 42.4 to 32.3 %). Our further study will focus on the influences of CO₂ partial pressure and carbonation temperature upon carbonation kinetics, and the effect of SO₂ and H₂O should be also considered.

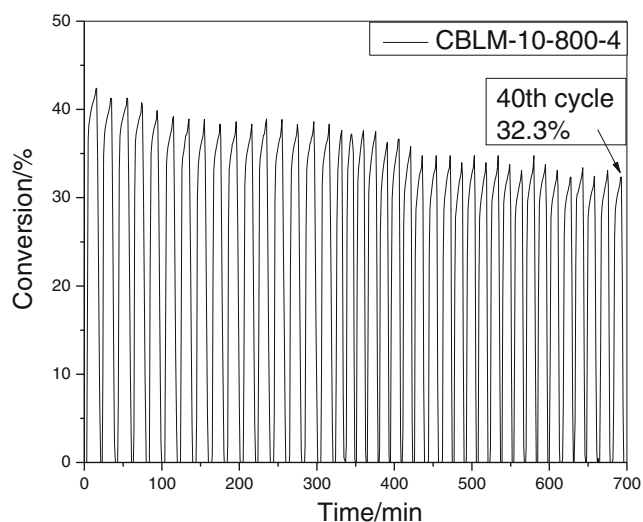


Fig. 7 Cyclic CO₂ capture conversion of CBLM-10-800-4 at 40 cycles in TGA (carbonation: 750 °C, 10 min, 15 % CO₂/85 % N₂; calcination: 750 °C, 5 min, 100 % N₂)

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Compliance with ethical standards

Conflict of interest The authors declare that they have no competing interests.

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