

Synthesis of highly reactive sorbent from industrial wastes and its CO₂ capture capacity

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Abstract: A kind of industrial solid waste, i. e., carbide slag, was used as CaO precursor to synthesize CO₂ sorbent. The highly reactive synthetic sorbent was prepared from carbide slag, aluminum nitrate hydrate and glycerol water solution by the combustion synthesis method. The results show that the synthetic sorbent exhibits a much higher CO₂ capture capacity compared with carbide slag. The CO₂ capture capacity and the carbonation conversion of the synthetic sorbent are 0.38 g/g and 0.70 after 50 cycles, which are 1.8 and 2.1 times those of carbide slag. The average carbonation conversion and the CO₂ capture efficiency of the synthetic sorbent are higher than those of carbide slag with the same sorbent flow ratios. The required sorbent flow ratios are lower for synthetic sorbent to achieve the same CO₂ capture efficiency compared with carbide slag. With the same sorbent flow ratio and CO₂ capture efficiency, the energy requirement in calciner for the synthetic sorbent is less than that for carbide slag.

Key words: carbide slag; synthetic CO₂ sorbent; CO₂ capture
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A great amount of industrial wastes are generated in industrial production every year, most of which are difficult to recover and are usually landfilled. If handled improperly, industrial wastes will pose a threat to human health, ground water resources and the atmospheric environment. It is of interest to convert those harmful solid wastes to harmless and useful materials^[1-3]. The research on the reuse of industrial wastes such as a substitute for limestone has been studied in different fields^[1, 4-5]. Some industrial solid wastes have been used to capture CO₂^[4-5]. Calcium looping, i. e., repetitive carbonation/calcination cycles of CaO, is a feasible CO₂ capture technology for fossil fuel-fired power plants^[6] and H₂ co-production^[7]. Utilization of industrial wastes such as CO₂ sorbent in calcium looping technology is a promising possibility how to reuse the industrial wastes in the view of circular economy

and sustainable development. Some industrial wastes such as carbide slag show favorable CO₂ capture capacity^[1]. However, the carbonation conversion of the industrial wastes decreases with the number of cycles, just like limestone. In order to efficiently capture CO₂ in fossil fuel combustion and H₂ production, it is necessary to improve the CO₂ capture capacity of industrial wastes.

Many methods to raise CO₂ capture capacity of calcium-based sorbent in the calcium looping cycles have been summarized^[8]. The CO₂ capture performances of the synthetic calcium-based sorbents prepared by dispersing CaO precursors across calcium aluminates (Ca₁₂Al₁₄O₃₃^[9], Ca₃Al₂O₆^[10], Ca₉Al₆O₁₈^[11], Ca₃Al₁₀O₁₈^[12]) as support materials have been investigated. The supporters can effectively stabilize the pore structure and increase the sintering resistance of the synthetic sorbents. The synthetic CO₂ sorbents containing various calcium aluminates are determined by the synthesis routes (the wet mixing method, precipitation method, the sol-gel-combustion-synthesis (SGCS) method and the template method), raw materials such as CaO precursor, Al₂O₃ precursor and dispersant^[9, 13]. However, the raw materials used in the existing process such as nano CaCO₃, calcium acetate, and calcium-naphthenate, Ca(C₆H₅O₇)₂ are costly and some solvents and dispersants such as 2-propanol, citric acid and xylene are not cheap. In this paper, we use a type of industrial solid waste, i. e., carbide slag, as CaO precursor and glycerol water as solvent to synthesize a new type of CO₂ sorbent. The synthetic CO₂ sorbent is prepared from carbide slag, aluminum nitrate hydrate and glycerol water solution by the combustion synthetic method. Carbide slag and aluminum nitrate hydrate can be dissolved in the glycerol water solution. The glycerol is highly combustible. The synthetic sorbent is synthesized by Ca²⁺ and Al³⁺ in the glycerol water solution by the combustion of the glycerol, so the combustion process of the solution is also the synthesis process of the synthetic sorbent. The glycerol as a byproduct is obtained from the preparation of the biodiesel fuel^[14] and carbide slag is also almost free. Thus, the prepared synthetic sorbent is low-cost. The CO₂ capture performance of the synthetic sorbent is investigated. The average carbonation conversion, CO₂ capture efficiency and energy requirement in calciner for the post-combustion CO₂ capture system using a syn-

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thetic sorbent are calculated.

1 Experimental

1.1 Sorbent preparation

The carbide slag (sieved to size < 0.125 mm) mainly composed of $\text{Ca}(\text{OH})_2$ was sampled from a chlor-alkali plant located in Shandong Province, China. The chemical components of the carbide slag were analyzed by X-ray fluorescence (XRF) as shown in Tab. 1. The aluminum nitrate hydrate (analytical grade $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) and the glycerol (analytical grade $\text{C}_3\text{H}_8\text{O}_3$) were also used as the materials in the preparation of the synthetic sorbent. First, 50 mL glycerol was dissolved in 50 mL deionized water which was stirred at 25 °C for 20 min, and then, the solution was heated from 25 to 80 °C. Secondly, 10 g carbide slag and some $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were added into the solution and stirred at 80 °C. The mass ratios of CaO derived from the carbide slag to Al_2O_3 derived from $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were 90:10 (defined as the best ratio by previous works^[15]). After the carbide slag and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were completely dissolved in the glycerol water solution, it was sent to a muffle furnace for the combustion synthesis process. Subsequently, the solution was combusted in the muffle furnace (800 °C) under air for 1 h. Synthetic sorbents were obtained after the combustion and sieved to size < 0.125 mm. The XRD result shows that only CaO and $\text{Ca}_3\text{Al}_2\text{O}_6$ are found in the synthetic sorbent, as shown in Fig. 1. It reveals that CaO reacts with all Al_2O_3 to generate $\text{Ca}_3\text{Al}_2\text{O}_6$.

Tab. 1 Chemical components in carbide slag %

W_{CaO}	W_{MgO}	W_{SiO_2}	$W_{\text{Al}_2\text{O}_3}$	$W_{\text{Fe}_2\text{O}_3}$	$W_{\text{Na}_2\text{O}}$	W_{TiO_2}	W_{others}	Loss on ignition
71.09	0.09	2.51	1.96	0.16	0.02	0.04	1.28	22.85

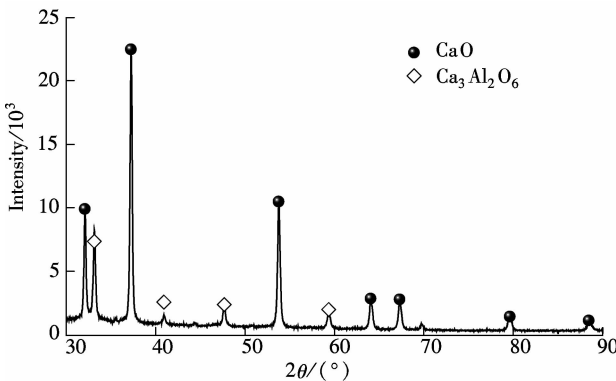


Fig. 1 XRD spectra of synthetic sorbent

1.2 Cyclic CO_2 capture tests

A dual fixed-bed reactor operated under atmospheric pressure, the details of which were given somewhere else, was used to determine the CO_2 capture behavior of the synthetic sorbents in the carbonation/calcination cy-

cles. The carbonation temperature was 700 °C, and the carbonation time was 30 min. The calcinations temperature was 850 °C, and the calcinations time was 10 min. Two parameters including CO_2 capture capacity C_N and carbonation conversion X_N were used to characterize the CO_2 capture performance of the synthetic sorbent as follows:

$$C_N = \frac{m_{\text{car},N}(t) - m_{\text{cal},N}}{m_0} \quad (1)$$

$$X_N = \frac{m_{\text{car},N}(t) - m_{\text{cal},N}}{m_0 c} \frac{M_{\text{CaO}}}{M_{\text{CO}_2}} \quad (2)$$

where t denotes the carbonation time, min; C_N is the CO_2 capture capacity of samples at t in the N -th cycle, which indicates CO_2 adsorption amount per unit mass of sample, g/g; X_N is the carbonation conversion of samples at t in the N -th cycle, which indicates the fractional conversion of CaO derived from sample to CaCO_3 , mol/mol; m_0 is the mass of initial sample, g; c is the mass percent of CaO in initial sample, %; $m_{\text{cal},N}$ is the sample mass after complete calcination in the N -th cycle, g; $m_{\text{car},N}(t)$ is the sample mass after carbonation at t in the N -th cycle, g; M_{CaO} and M_{CO_2} are the molar mass of CaO and CO_2 , respectively, g/mol.

1.3 Average carbonation conversion, CO_2 capture efficiency and energy requirement in calciner

An equation is employed to fit the carbonation conversion curve for the calcium-based sorbent as follows^[16]:

$$X_N = f_m^N (b - f_w) + f_w \quad (3)$$

where b , f_m and f_w are the fitting constants.

The CO_2 capture efficiency E_{CO_2} is defined as^[17]

$$E_{\text{CO}_2} = \frac{F_0 + F_R}{F_{\text{CO}_2}} X_{\text{ave}} \quad (4)$$

where F_0 is the makeup flow rate of fresh sorbent, kmol/s; F_R is the flow rate of recycled sorbent excluding fresh makeup, kmol/s; F_{CO_2} is the flow rate of CO_2 produced by coal combustion entering the carbonator, kmol/s; X_{ave} is the average carbonation conversion.

The average carbonation conversion is defined as

$$X_{\text{ave}} = \sum_{k=1}^{k=\infty} r_k X_k \quad (5)$$

where r_k is the mass fraction of CaO derived from the fresh calcium-based sorbent entering the carbonator in $F_0 + F_R$ (kmol/s) after k cycle; X_k is the carbonation conversion after k cycles.

The average carbonation conversion of the calcium-based sorbent can be described by

$$X_{\text{ave}} = \frac{F_0 f_m (b - f_w)}{F_0 + F_R (1 - f_m)} + f_w \quad (6)$$

and the CO₂ capture efficiency can be calculated by

$$E_{\text{CO}_2} = \frac{F_0 + F_R}{F_{\text{CO}_2}} X_{\text{ave}} = \frac{F_0/F_R + 1}{F_{\text{CO}_2}/F_R} \left(\frac{(F_0/F_R)f_m(b-f_w)}{F_0/F_R + 1 - f_w} + f_w \right) \quad (7)$$

The regeneration of CaO is an endothermic reaction, and the energy requirement in the calciner is an important parameter. The heat requirement for heating unreacted CaO, formed CaCO₃, Ca₃Al₂O₆ and the inert mass from carbonation temperature to calcinations temperature are defined as

$$H_1 = [c_{p,\text{CaO}} F_R (1 - X_{\text{ave}}) + c_{p,\text{CaCO}_3} F_R X_{\text{ave}} + c_{p,\text{inert}} F_{R,\text{inert}} + c_{p,\text{Ca}_3\text{Al}_2\text{O}_6} F_{R,\text{Ca}_3\text{Al}_2\text{O}_6}] \Delta T_{\text{calc-carb}} \quad (8)$$

where c_p is the heat capacity, J/kmol; $\Delta T_{\text{calc-carb}}$ is the temperature difference between the calciner and carbonator, K.

The heat requirement for the calcination of CaCO₃ formed during carbonation is calculated as

$$H_2 = F_R X_{\text{ave}} h_{\text{CaCO}_3} \quad (9)$$

where h_{CaCO_3} is the calcination reaction heat of CaCO₃, kJ/mol.

The fresh sorbent filled into the calciner is heated to the calcinations temperature from the ambient temperature and the heat requirement is calculated as

$$H_3 = (c_{p,\text{CaO}} F_0 + c_{p,\text{Ca}_3\text{Al}_2\text{O}_6} F_{0,\text{Ca}_3\text{Al}_2\text{O}_6} + c_{p,\text{inert}} F_{0,\text{inert}}) \Delta T_{\text{calc-fresh}} \quad (10)$$

where $\Delta T_{\text{calc-fresh}}$ is the temperature difference between the calciner and fresh sorbent, K.

According to Tab. 1, it is easily obtained that

$$F_{0,\text{Ca}_3\text{Al}_2\text{O}_6} = 0.077 F_0 \quad (11)$$

$$F_{R,\text{Ca}_3\text{Al}_2\text{O}_6} = 0.077 F_R \quad (12)$$

Incorporating Eqs. (8) to (10), the energy requirement H_{req} in the calciner is obtained as

$$H_{\text{req}} = H_1 + H_2 + H_3 \quad (13)$$

Then, the energy requirement per molar captured CO₂ is calculated as

$$\begin{aligned} \frac{H_{\text{req}}}{F_{\text{CO}_2}} = & \left[c_{p,\text{CaO}} \frac{F_R}{F_{\text{CO}_2}} (1 - X_{\text{ave}}) + c_{p,\text{CaCO}_3} \frac{F_R}{F_{\text{CO}_2}} X_{\text{ave}} + \right. \\ & \left. c_{p,\text{inert}} F_{R,\text{inert}} + 0.077 c_{p,\text{Ca}_3\text{Al}_2\text{O}_6} \frac{F_R}{F_{\text{CO}_2}} \right] \Delta T_{\text{calc-carb}} + \\ & \frac{F_R}{F_{\text{CO}_2}} X_{\text{ave}} h_{\text{CaCO}_3} + \left[c_{p,\text{CaO}} \frac{F_0}{F_{\text{CO}_2}} + 0.077 c_{p,\text{Ca}_3\text{Al}_2\text{O}_6} \frac{F_0}{F_{\text{CO}_2}} + \right. \\ & \left. c_{p,\text{inert}} F_{0,\text{inert}} \right] \Delta T_{\text{calc-fresh}} \quad (14) \end{aligned}$$

2 Results and Discussion

2.1 CO₂ capture performance of synthetic sorbent

Fig. 2 shows the CO₂ capture capacity and carbonation

conversion of the synthetic sorbent and carbide slag having undergone 50 cycles. It can be seen that the synthetic sorbent shows much higher CO₂ capture capacity and carbonation conversion compared with carbide slag. C_{50} and X_{50} of the synthetic sorbent are 0.38 g/g and 0.70, which are 1.8 and 2.1 times those of carbide slag. Also, the synthetic sorbent demonstrates the slight decay in CO₂ capture capacity and carbonation conversion with the number of cycles. The glycerol is soluble in the water. At the same time, carbide slag and Al(NO₃)₃ · 9H₂O can be dissolved in the glycerol water solution. Thus, the obtained solution is the homogeneous solution. Ca²⁺ and Al³⁺ are distributed uniformly in the synthetic sorbent, which is helpful for Ca₃Al₂O₆ to play a better role in the synthetic sorbent as support materials. On the other hand, CO₂ and water vapour are quickly released from the synthetic sorbent due to the rapid burning of the glycerol water solution at high temperatures, which can lead to the formation of the porous structure. This is a possible reason that glycerol addition enhances the cyclic CO₂ capture capacity of the synthetic sorbent.

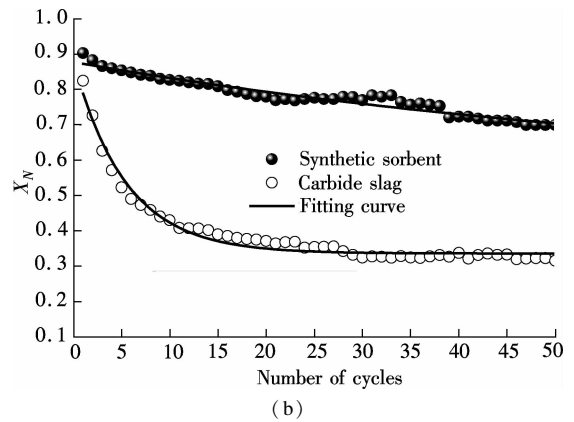
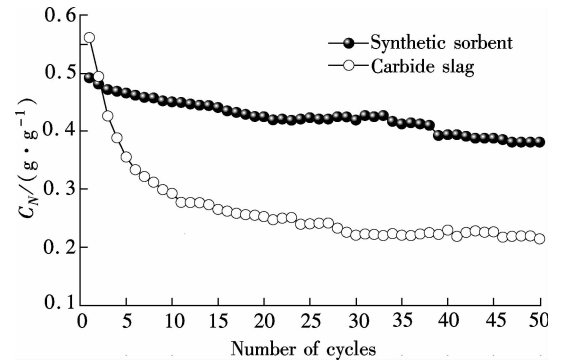


Fig. 2 CO₂ capture capacity and carbonation conversion of synthetic sorbent and carbide slag in 50 cycles (carbonation at 700 °C for 30 min in 15% CO₂, calcination at 850 °C for 10 min in pure N₂). (a) CO₂ capture capacity; (b) Carbonation conversion

2.2 Average carbonation conversion of synthetic sorbent and carbide slag

Eq. (3) is employed to fit the carbonation conversion

data of the synthetic sorbent and carbide slag. The fitting curves are shown in Fig. 2(b) and the fitting results are shown in Tab. 2. Then, the average carbonation conversion can be calculated according to Eq. (6), as shown in Fig. 3. The sorbent flow ratios, such as F_0/F_{CO_2} and F_R/F_{CO_2} , have a comparable effect on the average carbonation conversion. For both synthetic sorbent and carbide slag, X_{ave} increases with the increase of F_0/F_{CO_2} . When F_0/F_{CO_2} is a constant, X_{ave} decreases with the increase of F_R/F_{CO_2} . This is mainly because the carbonation conversion of recycled sorbent is lower than that of the fresh one. It can be seen that the X_{ave} of the synthetic sorbent is higher than that of carbide slag with the same F_0/F_{CO_2} and F_R/F_{CO_2} . For example, when $F_0/F_{CO_2} = 0.05$ and $F_R/F_{CO_2} = 1.5$, the X_{ave} of the synthetic sorbent is 0.80, which is 1.9 times that of carbide slag. For $F_R/F_{CO_2} = 1.5$, the X_{ave} of the synthetic sorbent is 0.67 when $F_0/F_{CO_2} = 0.01$, while the X_{ave} of carbide slag is only 0.54 when $F_0/F_{CO_2} = 0.2$.

Tab. 2 Fitting results of the carbonation conversion data of synthetic sorbent and carbide slag by Eq. (3)

Sample	b	f_m	f_w	R^2
Synthetic sorbent	0.88	0.99	0.53	0.936
Carbide slag	0.88	0.83	0.34	0.975

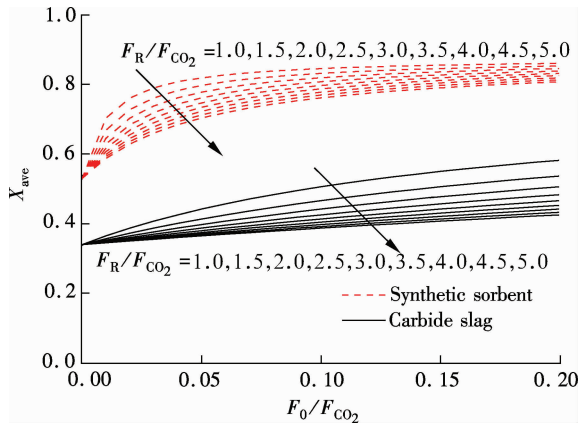


Fig. 3 Effect of flow ratios on average carbonation conversion of synthetic sorbent and carbide slag

2.3 CO₂ capture efficiency of synthetic sorbent and carbide slag

The effect of flow ratios, including F_0/F_{CO_2} and F_R/F_{CO_2} on CO₂ capture efficiency can be obtained according to Eq. (7), as shown in Fig. 4. E_{CO_2} increases clearly with the increase of F_0/F_{CO_2} and F_R/F_{CO_2} . The E_{CO_2} of the synthetic sorbent is higher than that of carbide slag with the same F_0/F_{CO_2} and F_R/F_{CO_2} . For example, when $F_0/F_{CO_2} = 0.05$ and $F_R/F_{CO_2} = 1.0$, the E_{CO_2} of the synthetic sorbent is 86.0%, and the E_{CO_2} of carbide slag is only 46.4%. The synthetic sorbent can achieve a higher CO₂ capture efficiency with the same F_0/F_{CO_2} and F_R/F_{CO_2} , compared with carbide slag. For $F_R/F_{CO_2} = 1.5$, the E_{CO_2}

of the synthetic sorbent is close to 100% when F_0/F_{CO_2} is 0.01. For carbide slag, the E_{CO_2} of 100% was achieved when F_0/F_{CO_2} is as high as 0.14 and F_R/F_{CO_2} is 2.0. The required F_0/F_{CO_2} and F_R/F_{CO_2} are lower for the synthetic sorbent to achieve the same E_{CO_2} compared with carbide slag.

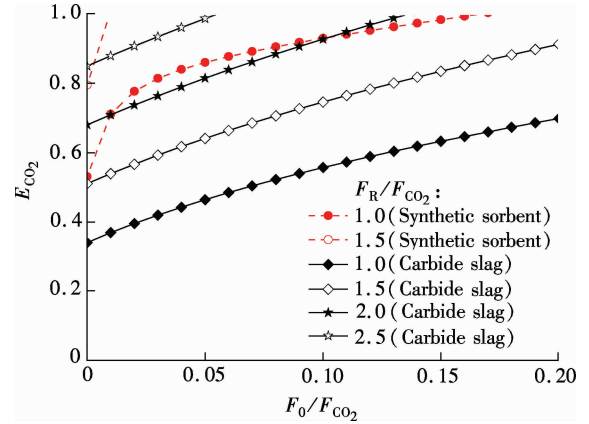


Fig. 4 Effect of flow ratios on CO₂ capture efficiency of synthetic sorbent and carbide slag

2.4 Energy requirement in calciner

For synthetic sorbent and carbide slag, increasing F_0/F_{CO_2} and F_R/F_{CO_2} means a higher CO₂ capture efficiency. However, higher F_0/F_{CO_2} and F_R/F_{CO_2} lead to an increase of energy requirement in the calciner. As shown in Fig. 5, for both synthetic sorbent and carbide slag, H_{req}/F_{CO_2} increase with the increase of F_0/F_{CO_2} and F_R/F_{CO_2} . Also, it has a linear relationship between H_{req}/F_{CO_2} and F_0/F_{CO_2} in the range of the calculated data. With the same F_0/F_{CO_2} and F_R/F_{CO_2} , the energy requirement for the synthetic sorbent is higher than that for carbide slag. That is mainly due to the higher concentration of CaCO₃ in the synthetic sorbent. Fig. 6 shows the effect of E_{CO_2} on energy requirement in the calciner. It can be seen that H_{req}/F_{CO_2} increases with the increase of E_{CO_2} , for both synthetic sorbent and carbide slag. With the same flow ratio and

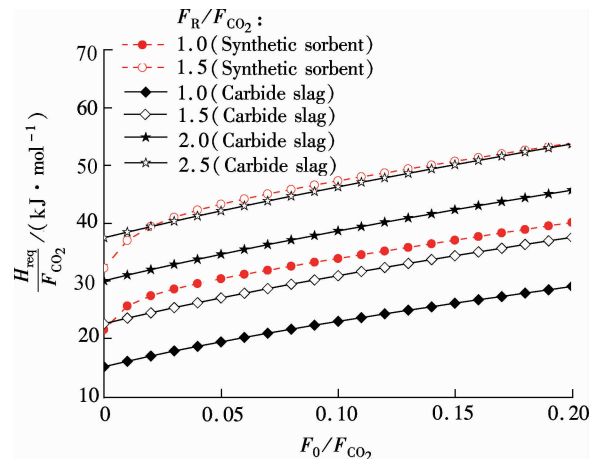


Fig. 5 Effect of flow ratios on energy requirement in calciner

E_{CO_2} , $H_{\text{req}}/F_{\text{CO}_2}$ for the synthetic sorbent is less than that for carbide slag. It reveals that less energy is required when using a synthetic sorbent as CO₂ sorbent in the calcium looping process.

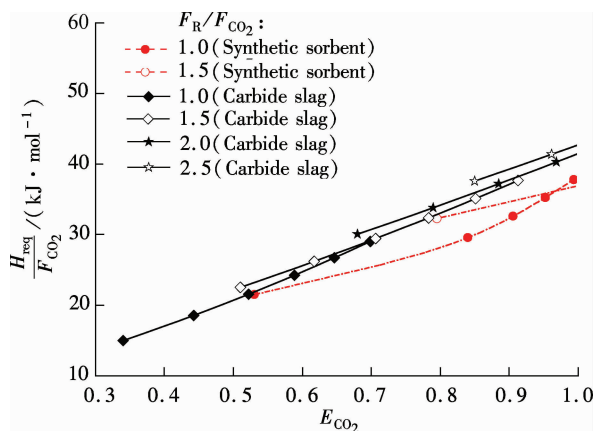


Fig. 6 Effect of E_{CO_2} on energy requirement in calciner

3 Conclusion

A type of highly reactive synthetic CO₂ sorbent is prepared from carbide slag, aluminum nitrate hydrate and glycerol water solution by the combustion synthesis method. The CO₂ capture performance of the synthetic sorbent is investigated. The average carbonation conversion, CO₂ capture efficiency and energy requirement in a calciner for the post-combustion CO₂ capture system using synthetic sorbent are calculated. The synthetic sorbent shows a much higher CO₂ capture capacity and carbonation conversion compared with carbide slag. The uniform distribution of Ca²⁺ and Al³⁺ in the synthetic sorbent and the formation of the porous structure due to the rapid burning of the glycerol water solution may be two possible reasons for the high cyclic CO₂ capture capacity of the synthetic sorbent. The X_{ave} and E_{CO_2} of synthetic sorbent are higher than those of carbide slag with the same F_0/F_{CO_2} and F_R/F_{CO_2} . The required F_0/F_{CO_2} and F_R/F_{CO_2} are lower for the synthetic sorbent to achieve the same E_{CO_2} compared with carbide slag. With the same sorbent flow ratio and E_{CO_2} , $H_{\text{req}}/F_{\text{CO}_2}$ for the synthetic sorbent is less than that for carbide slag. The synthetic sorbent is more suitable as a CO₂ sorbent in the calcium looping process compared with carbide slag.

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基于钙基废弃物的高活性吸收剂合成及其循环捕集 CO_2 性能

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摘要:利用一种典型的钙基废弃物电石渣作为 CaO 源合成 CO_2 吸收剂. 该高活性吸收剂由电石渣、九水硝酸铝和甘油水溶液经燃烧合成法合成. 结果表明, 该合成吸收剂表现出明显好于电石渣的 CO_2 捕集性能. 50 次循环后, 合成吸收剂的 CO_2 吸收量和碳酸化转化率为 0.38 g/g 和 0.70, 分别是电石渣相同循环次数时的 1.8 和 2.1 倍. 当吸收剂补充率相同时, 合成吸收剂的平均碳酸化转化率和 CO_2 捕集效率均高于电石渣. 为取得相同的 CO_2 捕集效率, 合成吸收剂所需吸收剂的补充率小于电石渣. 当吸收剂补充率和 CO_2 捕集效率相同时, 用合成吸收剂作为 CO_2 吸收剂时煅烧炉内所需的能量小于用电石渣作为 CO_2 吸收剂时所需能量.

关键词:电石渣; 合成 CO_2 吸收剂; CO_2 捕集

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