Effective Capture of Carbon Dioxide Using Hydrated Sodium Carbonate Powders

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Abstract: The emission of CO_2 has been considered a major cause of greenhouse effects and global warming. The current CO_2 capture approaches have their own advantages and weaknesses. We found that free-flowing hydrated sodium carbonate (Na_2CO_3) powders with 30 wt % water can achieve a very high CO_2 sorption capacity of 282 mg/g within 60 min and fast CO_2 uptake (90% saturation uptake within 16 min). The results suggest that the alkaline solution resulting from the dissolution of partial Na_2CO_3 can freely attach onto the hydrated Na_2CO_3 particles, which provides an excellent gas–liquid interface for CO_2 capture, leading to significantly enhanced CO_2 sorption capacity and kinetics.

Keywords: hydrated sodium carbonate; CO_2 capture; free-flowing powders

1. Introduction

Emission of CO_2 is identified as the main contributor to global climate change. Reducing the levels of CO_2 in the atmosphere has become a pressing issue worldwide, and capturing and sequestrating CO_2 as an option to decrease levels of CO_2 has been widely explored [1–4].

A number of promising materials for CO_2 capture were reported [5–10]. The best developed are probably aqueous amines [11,12], including monoethanolamine (MEA) [13,14] and diethanolamine (DEA) [15,16]. However, liquid amines have some serious disadvantages, including amine evaporation [17,18], corrosion to equipment [19], and high energy cost for regeneration [20,21]. A feasible way to reduce the corrosivity and the regeneration energy is to use supported amine adsorbents [22–26], but the raw materials are currently too expensive to be applied in large-scale industrial settings [27].

As an alternative to supported amine sorbents, alkali metal carbonates such as K_2CO_3 and Na_2CO_3 as solid sorbents have received wide attention with both high sorption capacity and low cost [28–32]. However, the main problem of using carbonates is their slow reaction kinetics [33–35]. Cooper and co-workers reported that dry K_2CO_3 solution (K_2CO_3 aqueous solution coated with hydrophobic silica powders) exhibited significantly increased CO_2 uptakes [36], but the recyclability of this sorbent was poor. It has been generally accepted that K_2CO_3 is superior to Na_2CO_3 in terms of both CO_2 uptake capacity and kinetics [37–39]. However, using Na_2CO_3 will be more competitive for

large-scale industrial applications because of its lower cost, especially if one can dramatically promote the rate of the key reaction:

$$Na_2CO_3 + H_2O + CO_2 \rightleftharpoons 2NaHCO_3 \qquad (1)$$

One of the most common approaches to tackle this problem is to disperse Na_2CO_3 powders on solid supports [40,41], but such a strategy also reduces CO_2 sorption capacity because the inclusion of the supports greatly decreases the amount of active components per unit mass [42].

In this report, we demonstrate that support-free hydrated sodium carbonate powders (HSCPs) prepared by simply mixing a certain amount of water and Na_2CO_3 powders exhibit effective CO_2 capture. The alkaline solution resulting from the dissolution of partial Na_2CO_3 can freely attach into hydrated Na_2CO_3 particles, which provides an excellent gas–liquid interface for CO_2 capture, leading to significantly enhanced CO_2 sorption capacity and kinetics. The elimination of supports not only reduces the overall cost of raw materials, but also increases the CO_2 sorption capacity, both of which are critical for large-scale applications.

2. Experimental

2.1. Preparation of HSCPs

Na_2CO_3 (99.8%) was purchased from Tianjin Qilun Chemical Technology Co. Ltd., Tianjin, China. $Na_2CO_3 \cdot H_2O$ (99%) was purchased from Aladdin Co. Ltd., Shanghai, China. MEA (99%) was purchased from Jiangsu Yonghua Chemical Technology Co. Ltd., Changshu, China. CO_2 (99.9%) was supplied by Zhuozheng Gas Co. Ltd., Guangzhou, China. All the chemicals were used as received without further purification. A series of HSCPs with different Na_2CO_3 contents were prepared by thoroughly mixing an appropriate amount of Na_2CO_3 and deionized water at room temperature.

2.2. Characterization

X-ray diffraction (XRD) patterns of the samples were recorded using a Bruker D8 diffractometer (Bruker, Karlsruhe, Germany) with Bragg–Brentano $\theta-2\theta$ geometry (20 kV and 5 mA), using a graphite monochromator with Cu Kα radiation.

To measure the CO_2 capture capacity of the HSCP samples, 5.0 g HSCP was charged into a 50 mL container, which was exposed to CO_2 using a balloon containing a sufficient amount of CO_2 gas (ca. 5 L with a pressure of ca. 1.05 bar). The amount of CO_2 captured by each HSCP sample was measured using a balance. A muffle furnace (Luoyang BSK Electronic Materials Co. Ltd., Luoyang, China) was used to regenerate the sorbents at 250 °C for 1 h, which was mixed with water to reform HSCPs.

3. Results and Discussion

Figure 1a shows the CO_2 uptake kinetic curves using various HSCPs (labelled as HSCP-X, where X is the mass percentage of Na_2CO_3 in the mixture) as a sorbent at 30 °C. It was found that HSCP-10 to HSCP-60 had a very low CO_2 sorption capacity (<32 mg/g of HSCP). The CO_2 uptake capacity rapidly rose to 156 mg/g when the mass fraction of Na_2CO_3 was increased to 65 wt %, i.e., HSCP-65, but it still suffered from low sorption kinetics. Further increasing mass fraction of Na_2CO_3 led to another significant increase in term of both sorption capacity and kinetics. At the optimum concentration of 70 wt % (i.e., HSCP-70), the CO_2 uptake capacity reached 282 mg/g within 60 min, and the t_{90} (the time to achieve 90% of this capacity) was only 16 min. This capacity is much higher than that of other Na_2CO_3-based CO_2 sorbents reported in the literature, which varies between 32 and 140 mg/g [43,44]. Although HSCP-75 achieved the highest capacity (286 mg/g), its CO_2 sorption rate was relatively slow and t_{90} was about 45 min.
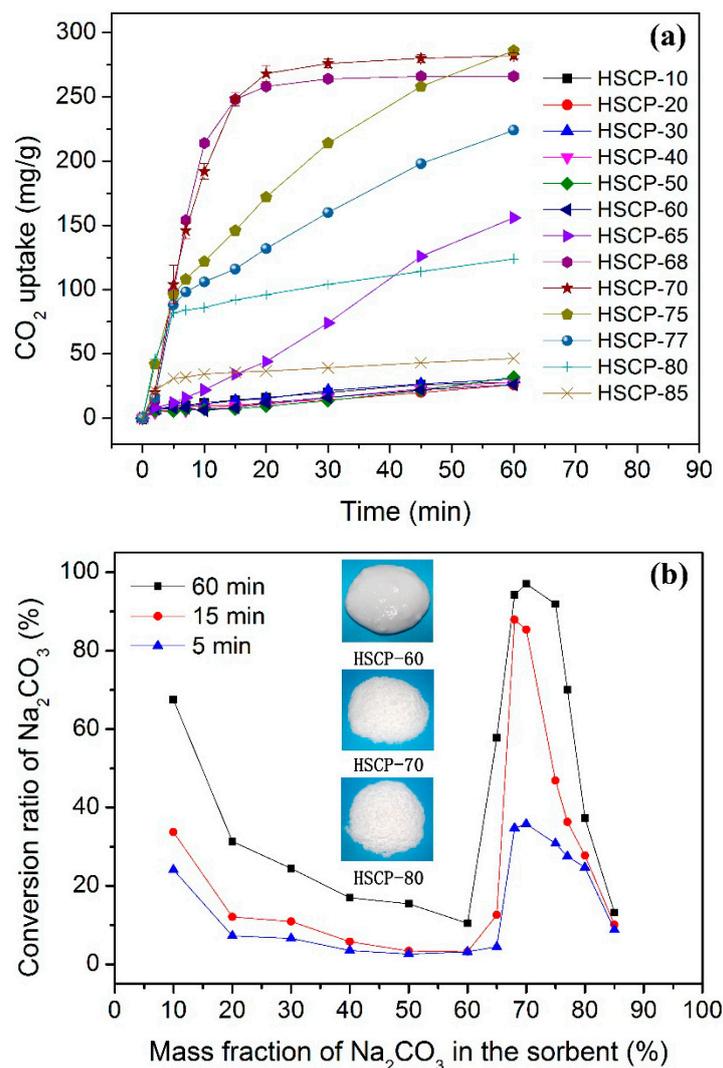
Figure 1. (**a**) CO_2 sorption kinetics of various HSCPs at 30 °C; (**b**) conversion ratio of Na_2CO_3 in various HSCPs and different reaction time.

It was found that too high a concentration of Na_2CO_3 in HSCP would actually lower the CO_2 uptake capacity. When the concentration of Na_2CO_3 in HSCP reached 80 and 85 wt %, the CO_2 uptake capacity decreased to 124 and 46 mg/g, respectively. Theoretically, the CO_2 sorption capacity is directly related to the amount of Na_2CO_3 in HSCPs when the content of water is more than 14.5 wt % according to Equation (1). Thus, the conversion ratio of Na_2CO_3 is a good indicator of the CO_2 sorption behaviour. As shown in Figure 1b, with an increasing mass fraction of Na_2CO_3, the conversion ratio of Na_2CO_3 decreased initially, then dramatically increased to a maximum value close to 100% before declining again. After 60 min of reaction, HSCP-70 exhibited the highest conversion rate (97.1%), which suggested that most of Na_2CO_3 was consumed. The HSCPs with a low mass fraction of Na_2CO_3, such as HSCP-10, also showed a high conversion ratio, in which Na_2CO_3 dissolved in water to form a solution, due to its high degree of hydrolysis [45]. However, their corresponding CO_2 uptake capacity is low because of the limited amount of Na_2CO_3 presented (Figure 1a). The other two sets of data in Figure 1b represent the conversion ratios of Na_2CO_3 after five and 15 min of reaction. For HSCP-70, its conversion ratio increased rapidly from 5 to 15 min, but changed little from 15 to 60 min, which suggested that most of Na_2CO_3 was consumed within 15 min, thus showing a high reaction rate.

Overall, the above results show that the concentration of Na_2CO_3 in HSCPs has a great influence on CO_2 capture, which can be explained by the fact that the morphology of HSCPs varies from aqueous solution and slurry, to powders with an increasing Na_2CO_3 concentration. At low Na_2CO_3 concentrations, the HSCPs exist as an aqueous solution or slurry as shown in Figure 1b (inset), which is not ideal for CO_2 capture because of the low gas–liquid contact surface area. However, HSCP-70 is a sample of free-flowing powders (Figure 2) with a much higher gas–liquid contact surface area. This is why it has a rapid reaction rate and a high CO_2 uptake capacity.

Figure 2. Free-flowing HSCP-70 from a glass funnel.

In order to better understand the mechanism of CO_2 sorption by HSCPs, the XRD patterns (Figure 3) of various Na_2CO_3-based compounds were collected, including the reaction products of HSCP-70 after 0, 5, 15, and 60 min of sorption reaction at 30 °C. The XRD pattern of HSCP-70 was very close to the standard pattern of $Na_2CO_3 \cdot H_2O$, which contains only 14.5 wt % water. This indicates that HSCP-70 contains extra water. As such, we also studied the CO_2 sorption by pure $Na_2CO_3 \cdot H_2O$, but it exhibited a low CO_2 sorption capacity and rate (Figure 4). This suggests that the extra water contained in the sorbent plays a significant role in CO_2 sorption. It indicates that the reaction proceeds most rapidly and effectively when Na_2CO_3, H_2O, and CO_2 are present simultaneously. Based on the above results, we propose that the extra water on the surface of HSCPs helps to form a basic alkaline aqueous environment. When CO_2 diffuses to the surface of HSCPs, it reacts with the basic aqueous media. Since the reaction is exothermic, the generated heat triggers the decomposition of sodium carbonate hydrates, meanwhile releasing water to drive the reaction to proceed continuously. In addition, along the reaction of HSCP-70 and CO_2, we also found that the characteristic peaks of Na_2CO_3 disappeared gradually, then intermediate structures, such as $Na_3H(CO_3)_2 \cdot 2H_2O$ (i.e., $Na_2CO_3 \cdot NaHCO_3 \cdot 2H_2O$) and $Na_2CO_3 \cdot 3NaHCO_3$, appeared after reacting for five and 15 min, respectively. Eventually, virtually pure $NaHCO_3$ formed after 60 min of reaction, which is expected.
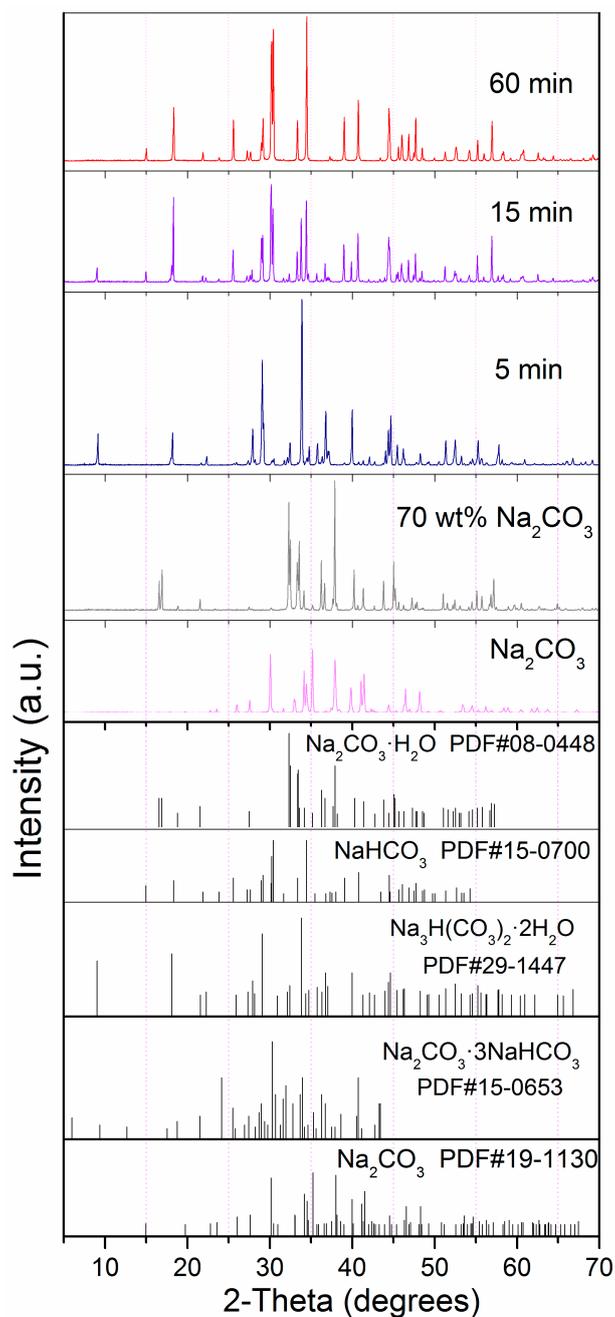
Figure 3. XRD patterns of various Na_2CO_3 based compounds and the reaction products of HSCP-70 after 0, 5, 15, and 60 min of CO_2 sorption reaction at 30 °C.

The amine-based CO_2 capture system is a proven technology that is already commercialized. To prevent excessive corrosion, typically 30 wt % MEA aqueous solution is used [11]. As shown in Figure 4, a 30 wt % MEA aqueous solution showed similar CO_2 uptake kinetics initially, but its overall sorption capacity was relatively low (111 mg/g versus 282 mg/g for HSCP-70). We also studied the CO_2 sorption capacity of pure water as a control, whose CO_2 uptake capacity was ca. 0.7 mg/g (Figure 4).
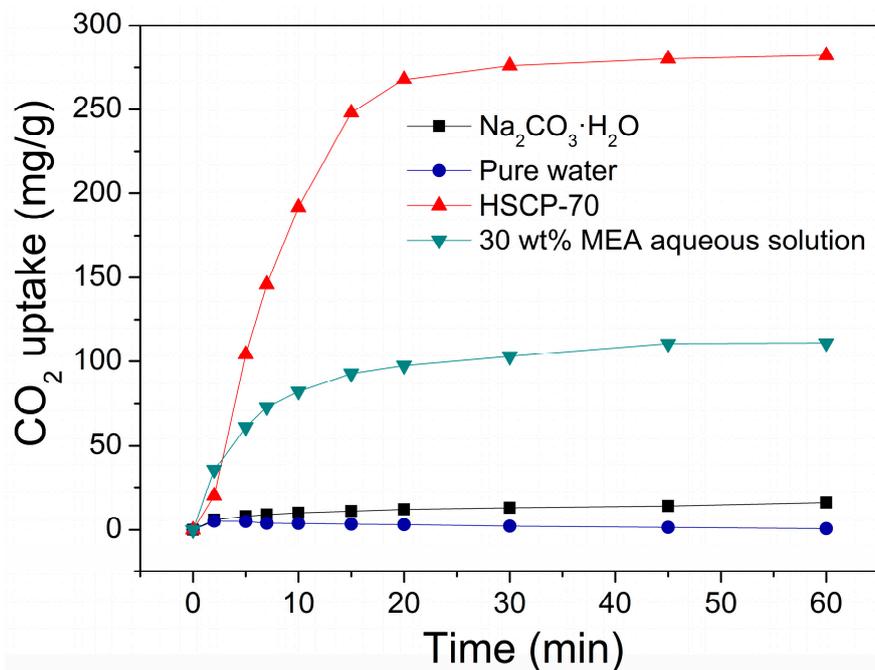
Figure 4. CO_2 sorption kinetics of HSCP-70, pure water, 30 wt % MEA aqueous solution, and $Na_2CO_3 \cdot H_2O$ at 30 °C.

We also studied the CO_2 uptake kinetics at different temperatures and the recyclability of HSCP-70. The suitable temperature range for CO_2 capture was determined to be 30–50 °C (Figure 5). A higher temperature will cause excessive evaporation of water in HSCP-70, and a lower temperature will cause the formation of $Na_2CO_3 \cdot 7H_2O$ (as shown in Figure 6), both of which lead to a lower CO_2 uptake of HSCP-70. HSCP-70 also exhibited excellent recyclability with little deterioration in CO_2 sorption capacity and reaction rate after recycling (Figure 7).
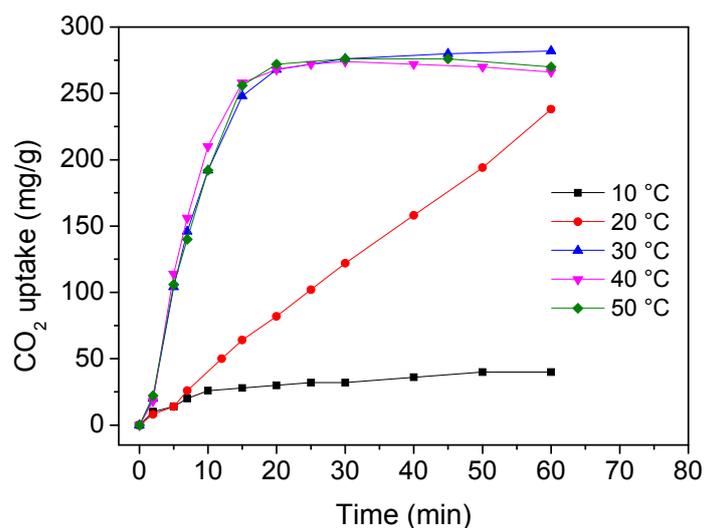

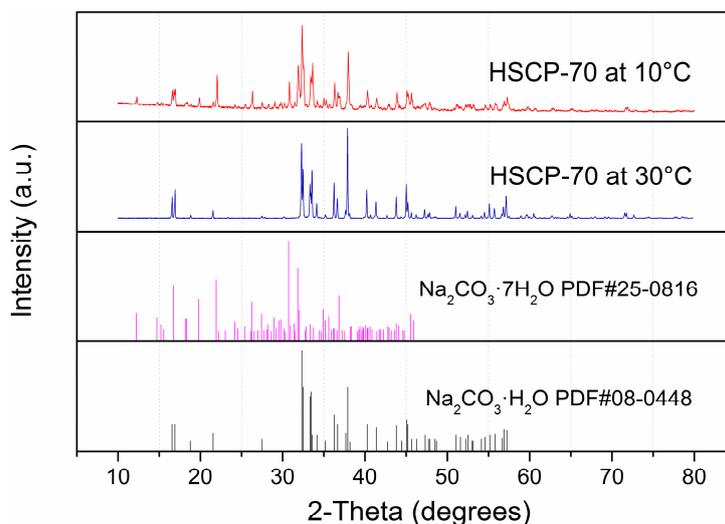**Figure 5.** CO_2 sorption kinetics of HSCP-70 at different temperatures.

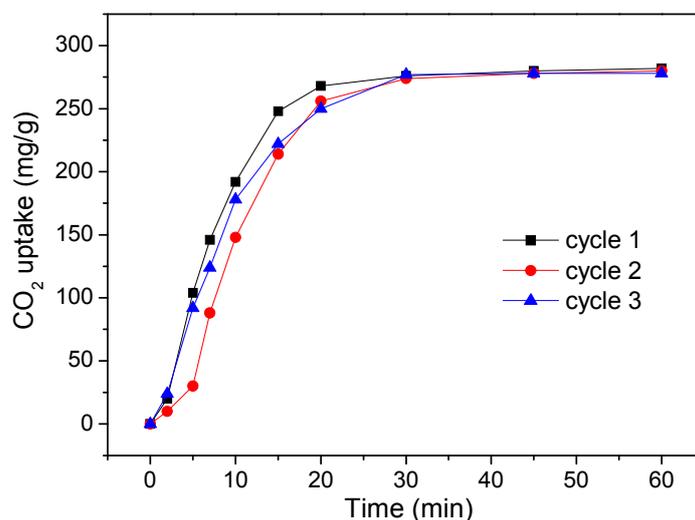**Figure 6.** XRD patterns of HSCP-70 at 10 and 30 °C.

Figure 7. Recycling performance of HSCP-70 after regeneration at 250 °C.

4. Conclusions

In summary, we have demonstrated that support-free HSCPs can be used as effective sorbents for CO_2 capture with a high capacity (282 mg/g) and fast sorption rate (90% saturation uptake within 16 min). The elimination of support and the low cost of Na_2CO_3 make this technology more competitive for large-scale applications. In addition, based on the reaction principles, HSCPs should also have high potential in capturing other acid gases, including SOx, NOx, H_2S, and Cl_2.

Author Contributions: Weixing Wang and Luyi Sun conceived and designed the experiments. Yuanhao Cai, Liang Li, Weilin Wang, Suying Wang, and Hao Ding performed the experiments and/or analyzed the data. Zhaofeng Wang, Zhengguo Zhang, and Hao Ding discussed the experiments and data. Weixing Wang, Luyi Sun, Yuanhao Cai, Weilin Wang, Zhaofeng Wang, and Hao Ding wrote the manuscript. All authors discussed the results and commented on the manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

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