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Performance of Extruded Particles from Calcium Hydroxide and Cement for CO₂ Capture

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ABSTRACT: The reversible reaction of CaO with CO₂ can be used for CO₂ capture. However, two challenging problems, i.e., loss-in-capacity and high attrition rate for CaO-based sorbents, must be solved before it can be practically applied. In this paper, sorbents with various CaO contents were prepared from calcium hydroxide and cement using a screw extruder and the physical and chemical properties of the sorbents were obtained. The mechanical properties of sorbent particles were tested by friability and compression testers, and the sorption capacity and regenerability were measured in a thermogravimetric analyzer. It appears that the sorbents occupied acceptable attrition resistance and good mechanical strength. In the meantime, the sorbent particles showed higher CO₂-capture capacities compared to pure CaO after 18 cycles, which was attributed to the stable phase of Ca₁₂Al₁₄O₃₃ in the sorbents. The sorbent particles would be suitable for the calcium looping processes.

1. INTRODUCTION

Global warming has been widely accepted to be caused by greenhouse gases, in particular, CO₂, and its atmospheric concentration has increased by ~35% to a value of 390 ppm since the industrial revolution.¹ The CO₂ concentration is projected to continue to rise if anthropogenic sources remain unchecked.² Therefore, the technologies that can effectively and economically reduce CO₂ emissions to the atmosphere are being actively developed around the world.

Currently, a relatively new method, calcium looping, is considered to be one of the promising technologies for CO₂ separation, owing to the high CO₂ sorption capacity (theoretically, 0.786 g of CO₂/g of CaO)^{3,4} and the abundant geological reserves of potential sorbents (approximately 4% of the earth's crust is comprised of calcium carbonate, which is a precursor for calcium-based sorbents).⁵

A calcium looping cycle is based on the reversible chemical reaction between CaO and CO₂; for example, CO₂ is captured by CaO in a carbonator to form CaCO₃, which is subsequently decomposed in a calciner to CaO and circulated back to the carbonator.^{6,7} The circulation between reactors would cause severe sorbent breakup and attrition.^{6,8,9} Additionally, the temperature difference between the reactors would lead to the rapid loss of the mechanical strength of the sorbent because of thermal stress. Therefore, the sorbents must have good mechanical properties to be used in the calcium looping process. Furthermore, to reduce cost, CaO-based sorbents should be repeatedly used for long cycles. However, there is a well-known problem of loss-in-capacity for CaO-based sorbents; i.e., the capacity of CaO-based sorbents decreases quickly after a number of carbonation/calcination cycles and eventually reaches an asymptotic value of 8–10% of its maximum theoretical value.⁴ Therefore, the sorbents must also have stable chemical properties without the problem of loss-in-capacity.

Until now, a number of methods have been proposed to attempt to overcome the problem of loss-in-capacity with varying degrees of success,^{4,10–16} such as thermal pretreatment,¹⁷

hydration,^{18–20} chemical modification,^{21,22} and synthesis of new sorbents with inert materials as support.^{23–25} However, to our knowledge, the work on enhancing the mechanical strength of CaO-based sorbents for CO₂ capture is still limited. Akiti et al.^{26,27} proposed a two-step method to fabricate core-in-shell pellets of calcium-based sorbents for desulfurizing hot coal gas. Cores and shells were both prepared with limestone and cement or alumina, but their contents were different to obtain pellets with a weak but highly reactive core encased in a strong and porous shell. Liu et al.²⁸ prepared some pellets with pure calcium carbonate and clay separately in cores and shells with a similar core-shell method. After three carbonation/calcination cycles, no decline in sorption capacity was found. Manovic et al.¹⁴ also fabricated sorbent pellets from limestone and cement, and a method of extrusion with sieve was used. When tested, the small pellets seemed to be strong, although some cracks and strength loss were observed by the large pellets after long-term carbonation/calcination cycles.

The present work was undertaken to investigate the feasibility of an alternative method to manufacture CaO-based sorbent particles using a screw extruder. Calcium hydroxide and commercial cement were chosen as the calcium precursor and strengthening material, respectively, for their low cost to synthesize economically attractive sorbents. The physical and chemical properties were investigated through mechanical testing and reactivity measurement to develop particles with high mechanical strength and acceptable CO₂ sorption capacity, thus, suitable for the calcium looping process.

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Table 1. Composition of the Commercial Hydrated Lime

component	Ca (OH) ₂	Mg (OH) ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	water
percentage (wt %)	90–95	0.5–1.0	0.5–3	0–2	0–0.4	0.1–2.5

Table 2. Composition of the Cement

component	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	TiO ₂	MgO	SO ₂	K ₂ O + Na ₂ O
percentage (wt %)	≤37.0	≥39.8	≤6.0	≤18.5	<4.0	<1.5	<0.4	<0.4

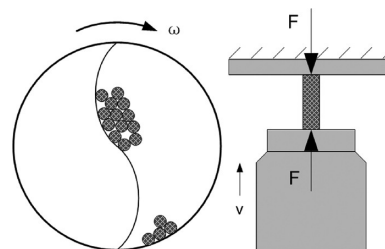
2. EXPERIMENTAL SECTION

2.1. Raw Materials and Sorbent Preparation. Two calcium hydroxides, reagent Ca(OH)₂ (96%) from Ajax Finechem and commercial hydrated lime (90–95%) from Cement Australia, and one cement produced by Kerneos Aluminate Technologies were used to synthesize CaO-based sorbents. The content of CaO in the sorbents varies from 25 to 75 wt %, and in total, eight sorbents were prepared. The synthesized sorbents were denoted as HC (if commercial hydrated lime was used as the calcium precursor) or CC (if reagent calcium hydroxide was used as the calcium precursor). For example, a sorbent with 25 wt % CaO derived from commercial hydrated lime and cement is designated as HC25, and the others are named in the same way. The compositions of commercial hydrated lime and cement are shown in Tables 1 and 2, respectively.

A PRISM EUROLAB 16 twin-screw extruder from Thermo Electron Corporation was used in this work, and the procedure for producing sorbent particles is as follows: Predetermined Ca(OH)₂ and cement in the desired ratio were physically mixed, and then the mixture was extruded to long cylinders through the extruder with a die (which has two holes, with the diameter of a hole being 2 mm). During the extrusion process, some water was added to assist solidification of the mixture and to improve the hardness of the particles. All sorbent particles were subjected to a chemical performance test after they were dried overnight at 110 °C and calcined at 900 °C for 3 h.

2.2. Sorbent Testing. The methods available to measure attrition of fluidized solids usually follow this principle: the weight percentage of particles reduced to less than a certain size in a specified time within a specified test apparatus may be used as an index representing the attrition propensity of the particulate solids.^{29,30} Therefore, here, attrition of the extruded particles was tested using a Friability Tester (25 rpm) from Vanderkamp with a vankel-type USP drum (inner diameter of about 28.5 cm). Before testing, sample particles (cylinders with a diameter of 2 mm) were cut to short cylinders with a length of about 1 cm and then the attrition tester was operated for 2000 and 4000 rotations. The weight of particles passing through a sieve with square apertures of 1 mm was taken as the mass loss caused by attrition. In addition, the compressive strength of extruded particles was measured using an Instron Universal Tester (type 4505). The particle (with a length of 3 mm) was placed between two plates, then force was gradually increased until the particle was broken. In this work, the compressive strength is defined as the ratio of the maximum breaking force to the original cross-sectional area of the particle. Every sample was measured at least 3 times, and the average value was finally adopted. The principles of attrition and compression tests are shown in Figure 1.

The CO₂ adsorbing capacities of the sorbents were determined with a Cahn thermogravimetric analyzer (TGA, model 121) using about 40–80 mg of sorbent on a quartz pan, which was suspended in a quartz tube. During testing, the temperature, time, and gas flow were controlled by Cahn WinTGA software. The sample mass was

**Figure 1.** Principle of mechanical property testing: (left) attrition test and (right) compression test.**Table 3. Weight Loss of Extruded Particles during Attrition Tests before and after Calcination**

sample	weight loss before calcination (%)		weight loss after 3 h of calcination at 900 °C (%)	
	after 2000 rotations	after 4000 rotations	after 2000 rotations	after 4000 rotations
HC25	3.55	5.45	4.58	8.01
HC40	4.28	5.64	9.14	12.10
HC50	4.60	6.10	10.96	15.39
HC75	2.21	3.30	10.35	15.19
CC25	6.78	8.64	8.20	11.78
CC40	7.12	9.76	11.27	15.03
CC50	8.06	11.30	10.66	14.60
CC75	5.36	7.69	7.66	11.28

monitored, and conversion was calculated on the basis of mass change, assuming that mass change was only caused by the formation and decomposition of calcium carbonate. The conditions used for one carbonation/calcination cycle are as follows, and it would be repeated for a desired number of cycles: First, the temperature of the TGA was raised to 900 °C at a rate of 20 °C/min under a nitrogen flow of 85 mL/min, and this temperature was kept for 10 min. Then, the sample was cooled to 650 °C at a rate of −20 °C/min. Once 650 °C was reached, a 15 mL/min flow of carbon dioxide was introduced into the TGA (so that the CO₂ concentration was 15 vol %), and this condition was maintained for 30 min for CO₂ sorption.

Phase composition of the sorbent was analyzed in a Bruker AXS D8 Advance X-ray diffractometer equipped with a scintillation counter, graphite monochromator, and copper target, and traces were processed using the Diffrac^{plus} Evaluation Package Release 2009 and PDF-2 2009.

The morphologies of samples were observed with a JEOL JSM-6610 scanning electron microscope (SEM) before and after multiple carbonation/calcination cycles. The particle was first crushed to expose its inner parts to the electron beam. Then, the powders were dispersed on the surface of conductive adhesive carbon tab and platinum-coated using an EIKO IB-5 Sputter Coater for 5 min (approximately 15 nm thick) to obtain a better electronic signal. All SEM images were obtained from secondary electrons with 20 kV of accelerating voltage.

3. RESULTS AND DISCUSSION

3.1. Physical Properties of Sorbents. Table 3 presents attrition testing results of the extruded samples with different CaO contents in the form of weight loss. Generally, HC and CC show similar attrition resistance, although HC appears to have less weight loss than CC before calcination.

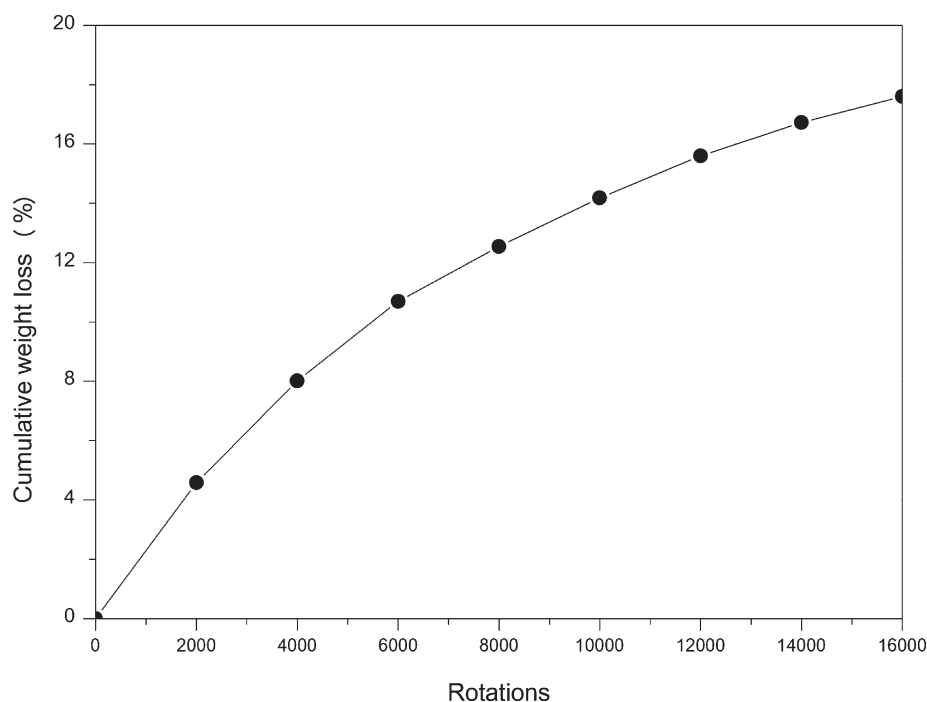


Figure 2. Weight loss of HC25 versus the number of rotations.

Table 4. Compression Test Results of Extruded Particles

sample	maximum compressive stress (MPa)			sample	maximum compressive stress (MPa)		
	before calcination	after 3 h calcination	after 18 cycles		before calcination	after 3 h calcination	after 18 cycles
HC25	28.40	22.50	22.34	CC25	21.42	13.02	10.35
HC40	23.58	10.32	7.43	CC40	10.07	5.83	5.01
HC50	16.84	6.07	5.14	CC50	6.21	3.93	4.90
HC75	15.42	5.14	4.53	CC75	8.04	<i>a</i>	<i>a</i>

^a Because cracks were found on the surfaces of some CC75 particles after calcination, no compressive test was carried out further on this sample.

Also, it can be seen that the attrition resistance of particles is not directly related to the content of the binder because the biggest weight loss occurs when about 40–50 wt % CaO is contained. Moreover, calcination has a severe negative influence on the attrition resistance of particles, which is undesirable for sorbent particles to be used in a real process. Before calcination, a weight loss of 5.45% for HC25 is observed after 4000 rotations; however, this weight loss rises to 8.01% after calcination. Among all, HC75 is most severely influenced by calcination, for which almost 4 times more weight loss after calcination is found. After 3 h of calcination and attrition test of 4000 rotations, HC25 demonstrates the smallest mass loss and has the best attrition resistance among all samples.

The weight loss tendency for a much longer attrition time is investigated for sample HC25, as shown in Figure 2. The weight loss grows with the number of rotations, but the attrition rate declines. For example, after the first 2000 rotations, about 4.58 wt % weight loss is observed; however, this value reduces to 1.85 and 0.87 wt % during the fourth and eighth 2000 rotations, respectively. This change is thought to be caused by the rounding effect on cylindrical particles prepared in this work. Later, the attrition rate gradually becomes lower, and it seems

that the weight loss is approaching a residual value of about 20% for HC25.

The extruded cylinders were also tested to determine their compressive strengths using a typical compression tester, and the results are presented in Table 4. Each sample is tested in three cases: before calcination, after calcination of 3 h, and after 18 cycles of carbonation/calcination. It is clear that the maximum compressive stress of extruded cylinders is bigger when more cement is contained in samples (28.40 MPa versus 15.42 MPa of HC25 and HC75 before calcination), which however does not correlate directly with the attrition data, as shown in Table 3.³¹ Additionally, all samples prepared with commercial hydrated lime show better compressive strength, in comparison to the samples derived from reagent calcium hydroxide when the content of calcium oxide is the same.

The maximum compressive strength declines when extruded particles are calcined, which is similar to the result of the attrition test. Interestingly, no apparent decrease of the compressive strength has been observed when particles are subjected to 18 cycles of carbonation/calcination. A reduction of less than 3 MPa is found for most sorbent particles; CC50 even presents a small increase, i.e., from 3.93 MPa before reaction to 4.90 MPa after 18

cycles. One possible explanation is that high-temperature calcination during preparation causes partial sintering of the sorbent, which helps to maintain the strength of the particle.

Although CC75 exhibits less weight loss than CC25 during the attrition test, it only shows a maximum compressive strength of 8.04 MPa before calcination. In contrast, the value is 21.42 MPa for CC25 under the same condition. Additionally, fragmentation occurs, and cracks are found on the surface of some calcined CC75 extruded particles, as shown in Figure 3c, which is very different from other samples; no cracks have been observed after calcination even after 18 carbonation/calcination cycles.

3.2. Chemical Properties of Sorbents. The CO_2 sorption capacities of the sorbents were tested using a TGA (model 121). On the basis of previous work,³² there would be some reactions between cement and calcium hydroxide during sorbent calcination. However, it is difficult to determine how much active CaO has been consumed. Therefore, in this work, the conversion values were calculated on the basis of the overall CaO content in calcium precursors. For this reason, the real carbonation

conversion should be higher than the reported data if the amount of consumed CaO is taken into account. Figure 4a shows the variation of the CaO conversion with the cycle number for six sorbents with three different CaO contents (25, 40, and 75 wt %). Their sorption capacities on grams of CO_2 per gram of sorbent basis were presented in Figure 4b.

There is a fast sorbent activity loss for all of the sorbents with an increasing cycle number, which is believed to be caused by sintering. However, the rate of reactivity loss is different for each sample. Pure $\text{Ca}(\text{OH})_2$ shows an initial conversion level of 59% and about 36% after 18 cycles. In contrast, despite a lower initial conversion level of 54%, which is likely related to the formation of mayenite, CC25 still has a conversion level of 45% after 18 cycle, a 9% decay of conversion, as compared to 23% decay for reagent $\text{Ca}(\text{OH})_2$. It also should be noted that, with an increasing cement content, the loss-in-capacity of CaO seems to be less severe during sorption/desorption cycles. For instance, CC25 exhibits a higher conversion level of 45% after the 18th cycle

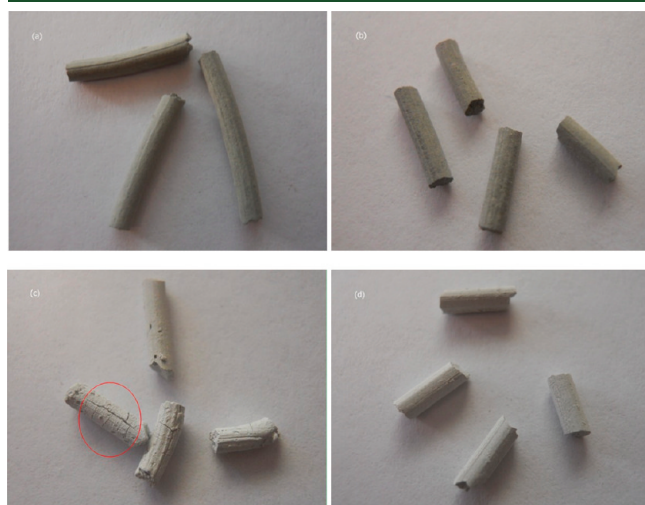


Figure 3. Photographs of (a) HC25 after 3 h of calcination, (b) HC25 after 18 cycles, (c) CC75 after calcination, and (d) HC75 after 18 cycles.

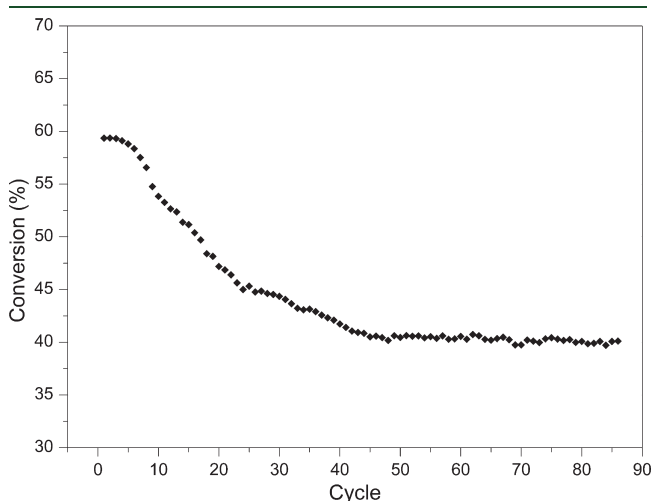


Figure 5. CaO conversion of CC25 particles for 86 carbonation/calcination cycles. Conditions: carbonation in 15 vol % CO_2 at 650 °C for 30 min and calcination in 100% N_2 at 900 °C for 10 min.

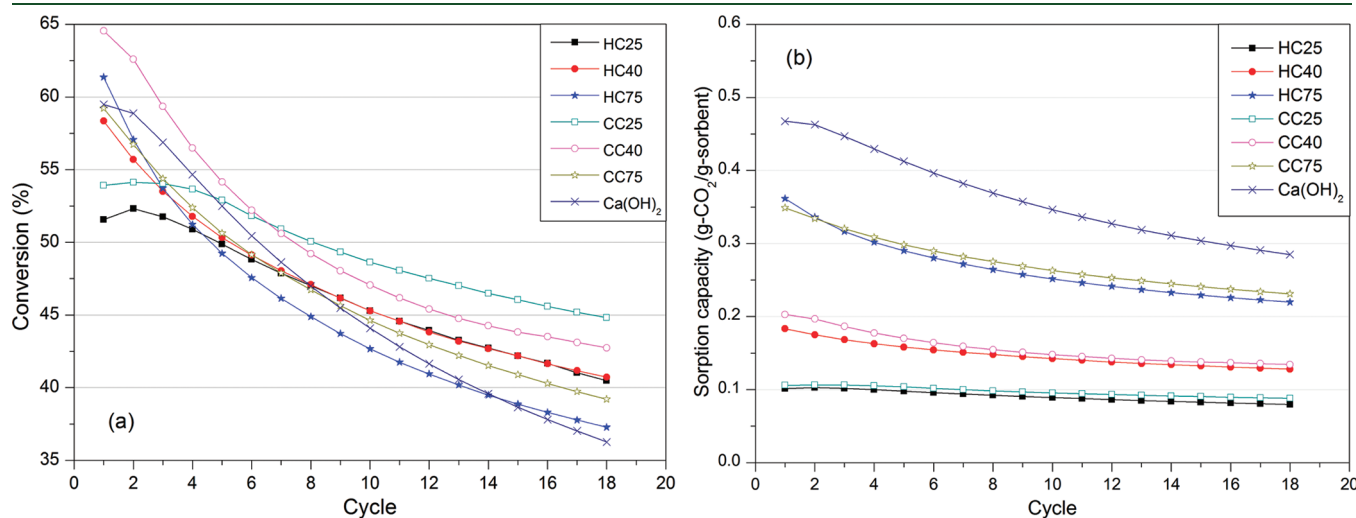


Figure 4. (a) Carbonation conversions and (b) sorption capacities of the extruded particles. Conditions: carbonation in 15 vol % CO_2 at 650 °C for 30 min and calcination in 100% N_2 at 900 °C for 10 min.

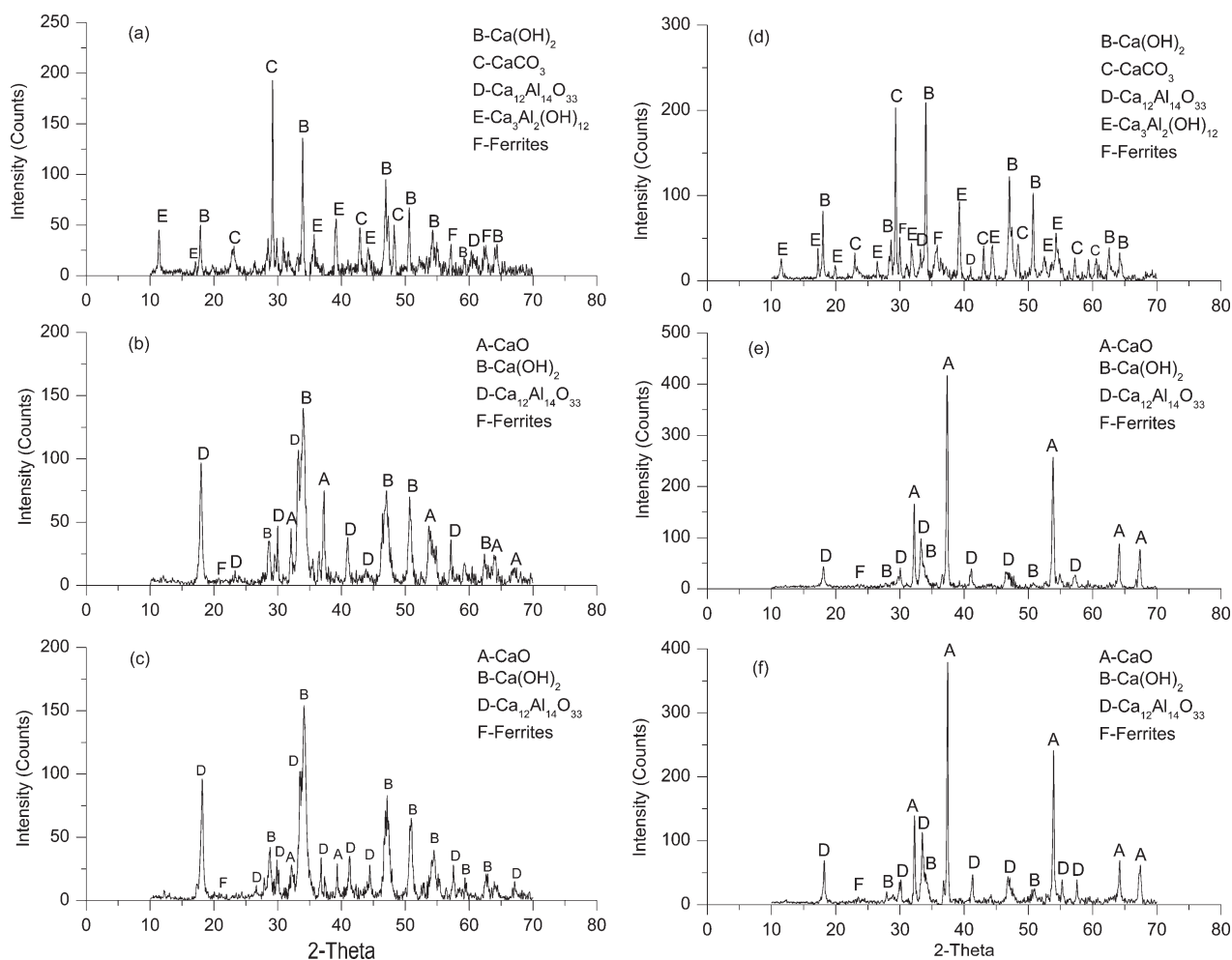


Figure 6. XRD patterns of (a) HC40 before calcination, (b) HC40 after calcination, (c) HC40 after 18 cycles, (d) CC40 before calcination, (e) CC40 after calcination, and (f) CC40 after 18 cycles.

compared to 39% for CC75. The HC sorbents show similar results. Additionally, the CC sample always has a slightly higher capacity than HC when they have the same CaO content. This is believed to be caused by the different contents of impurity in their calcium precursors, because when the impurity (4% for reagent calcium hydroxide and 5–10% for commercial hydrated lime) is taken into account, their carbonation conversions are almost the same. Figure 5 presents the sorption performance of CC25 particles for 86 carbonation/calcination cycles. Although a quick loss of capacity is observed for the initial ~46 cycles, the capacity seems to be maintained at a constant value of ~40% for the rest of the 40 cycles.

3.3. Characterization of Sorbents. As already mentioned above, all extruded particles demonstrated higher conversions than the CaO derived from pure Ca(OH)_2 after 18 carbonation/calcination cycles; therefore, we further investigated the composition and morphology of the sorbents to attempt to find the reasons. The XRD patterns of selected samples HC40 and CC40 are shown in Figure 6. All of the synthetic sorbents have similar composition after 3 h of calcination, and only CaO, $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$, and ferrites are observed. Ca(OH)_2 was observed to be present in some sorbents, as shown in panels b, c, e, and f of Figure 6, which was attributed to the hydration of CaO before XRD testing. Produced $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ is believed to act as an inert support and would not take part in

reactions during carbonation/calcination cycles, which could help reduce the rate of calcium aggregation during calcination.^{13,33} Therefore, the extruded particles with cement as the support precursor and binder show less severe loss-in-capacity compared to pure Ca(OH)_2 .

The problem of loss-in-capacity has been proposed to be attributable to grain growth and porosity decrease caused by sintering during calcination. Here, the morphologies of some selected sorbents before and after carbonation/calcination cycles are obtained and shown in Figure 7. The grain size of calcined Ca(OH)_2 is about $0.5\ \mu\text{m}$, and abundant pores are observed to be uniformly distributed around the crystals. After 18 cycles, aggregation of the crystal is observed and the grains grow to be much bigger than $2\ \mu\text{m}$. Simultaneously, small pores disappear, and big cracks are found, as shown in panels a and b of Figure 7. In comparison, the grains of CC40 and HC40 are a little smaller (around $0.2\ \mu\text{m}$) and the change of crystals and pores after 18 cycles is less pronounced. Therefore, the main reason for the higher carbonation conversion and better stability of the synthetic sorbents is believed to be due to the good resistance to morphology and structure change.

3.4. Effect of Crushing on Capacity. Although some improvement in CaO conversion has been achieved, as discussed

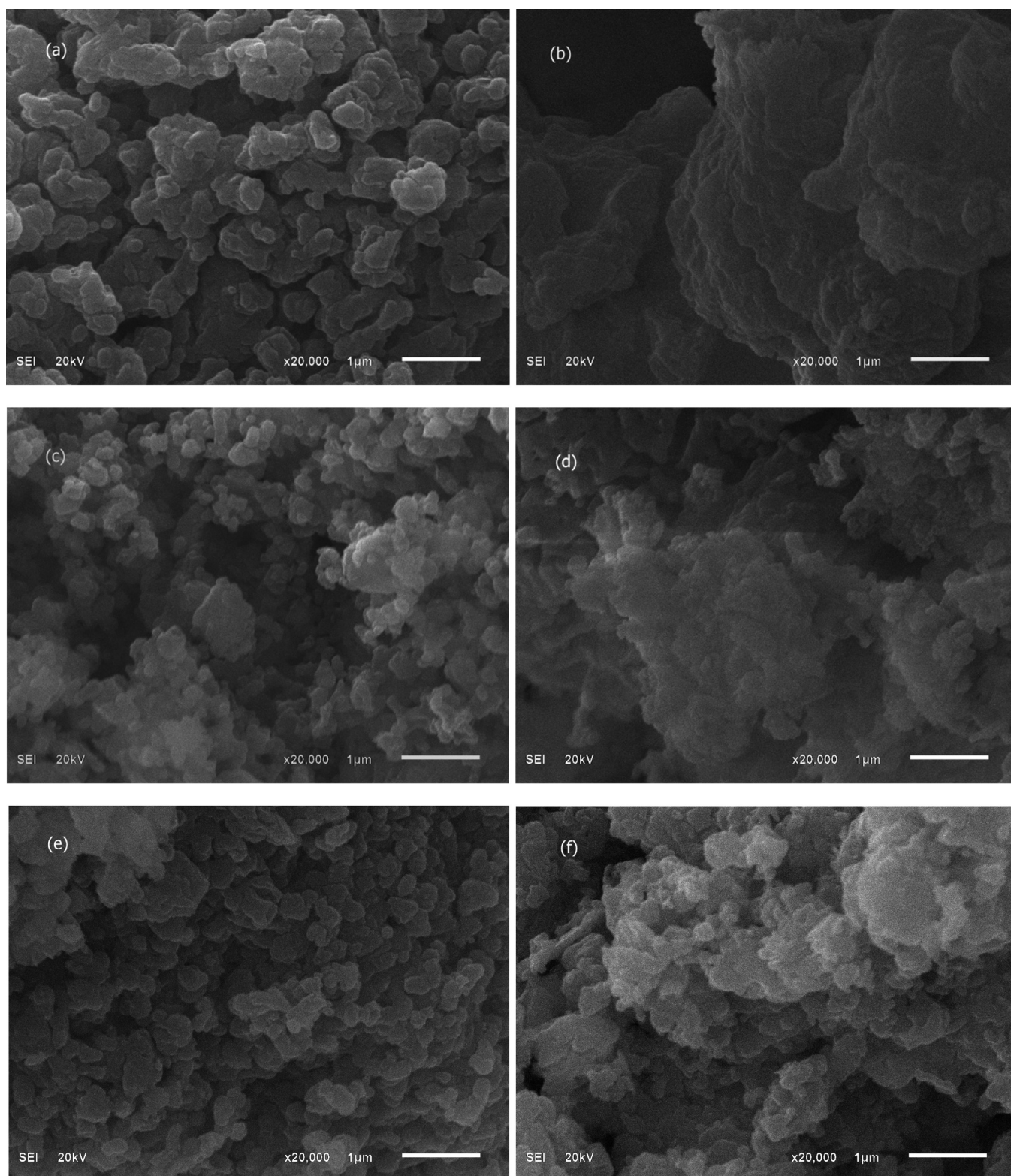


Figure 7. SEM images of selected sorbents: (a) $\text{Ca}(\text{OH})_2$ before cycles, (b) $\text{Ca}(\text{OH})_2$ after 18 cycles, (c) CC40 before cycles, (d) CC40 after 18 cycles, (e) HC40 before cycles, and (f) HC40 after 18 cycles.

previously, the adsorption enhancement is not as good as expected. To check the impact of particle manufacture on carbonation conversions, the extruded particles of CC25 and HC25 were crushed using a mortar to fine powder before

sorption/desorption cycle tests were carried out. The sorption performance curves of CC25 and HC25 after crushing are shown in Figure 8. Some differences can be seen before and after crushing of the particles; for example, the CaO conversion of

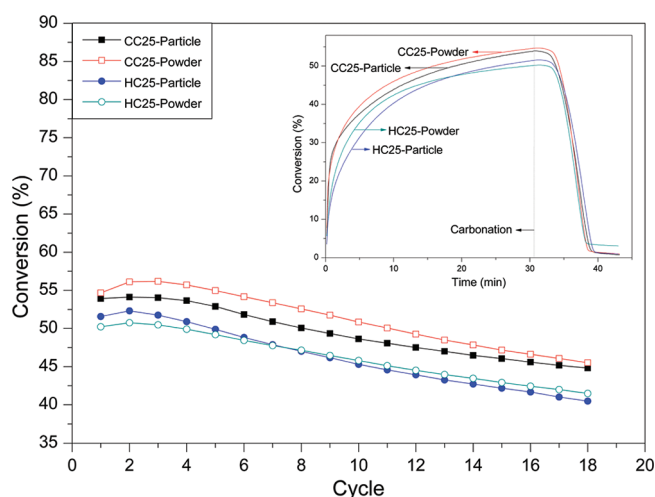


Figure 8. CaO conversion of original extruded particles and crushed powder. Conditions: carbonation in 15 vol % CO₂ at 650 °C for 30 min and calcination in 100% N₂ at 900 °C for 10 min. The inset shows the profile of the first carbonation.

the crushed CC25 is always higher than its original particles during 18 cycles. However, the difference in conversion is negligible because the maximum conversion difference is only around 2%, which indicates that the effect of the particle size and, thus, diffusional effect in the sorbent is negligible under the conditions studied.

3.5. Discussion. The use of the screw extruder for the manufacture of CO₂ sorbent particles has not been reported before. This work demonstrates that the sorbent particles prepared using a screw extruder have reasonable mechanical and chemical properties. The method can be easily scaled up for large-scale production of sorbent particles, although extra work is still needed to further improve the physical and chemical properties of the sorbents. It is also of interest to compare the performance of the sorbents produced using screw extrusion to that of those sorbents prepared using other techniques, such as core-in-shell.

Cement acts as both a binder and an inert support material in the sorbent particles. Improvement of CaO conversion has been observed for all of the sorbents with the addition of cement, because produced stable Ca₁₂Al₁₄O₃₃ can help prevent a dramatic structure change of the sorbent. However, the major advantage of the extruded sorbent particles would be the less attrition rate, which is highly desirable in practical operation. It appears that the sorbents prepared and tested in this work would be suitable for the calcium looping process, but higher capacity will be more beneficial. More work is being undertaken to further improve the capacity and stability of the sorbents by combining hydrated lime and cement together with other potential inert support material, such as a magnesium or aluminum precursor. Also, it would be more helpful for practical application if the chemical and mechanical performances of the extruded sorbent particles can be tested under actual conditions.

4. CONCLUSION

CaO-Based sorbent particles were manufactured via a method of screw extrusion with calcium hydroxide and cement as precursors. Moreover, the influence of the precursor type and

their content on the mechanical strength and chemical performance of the extruded particles was studied. It was found that screw extrusion is a potential method to prepare sorbent particles with good attrition resistance and mechanical strength. Commercial cement is a potential candidate to act as a binder to fabricate particles and also as a support material to enhance the long-term carbonation conversion of CaO-based synthetic sorbents. Additionally, the particle manufacture process has no effect on the chemical performance of CaO-based sorbents under the testing conditions.

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