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## Effect of cetyl trimethyl ammonium bromide concentration on structure, morphology and carbon dioxide adsorption capacity of calcium hydroxide based sorbents

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### ABSTRACT

Calcium hydroxide (Ca(OH)<sub>2</sub>) has been proposed as an important material for industrial, architectural, and environmental applications. In this study, calcium acetate was used as a precursor and cetyl trimethyl ammonium bromide (CTAB) was used as a surfactant to synthesize Ca(OH)<sub>2</sub> based adsorbents for carbon dioxide (CO<sub>2</sub>) capture. The effect of CTAB concentration (0.2–0.8 M) on the structure, morphology and CO<sub>2</sub> adsorption performance of Ca(OH)<sub>2</sub> was studied in detail. The synthesized samples were characterized by X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, field emission scanning electron microscopy (FESEM), BET surfaced area and thermogravimetry-differential thermal analysis (TG–DTA) techniques. The phase purity, crystallite size, Brunauer–Emmett–Teller (BET) surface area and CO<sub>2</sub> adsorption performance of Ca(OH)<sub>2</sub> precursor adsorbents were significantly increased when the concentration of 0.8 M. TGA results exhibited that 0.8 M of CTAB-assisted Ca(OH)<sub>2</sub> precursor adsorbent possessed a residual carbonation conversion of ~56% after 10 cycles.

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#### 1. Introduction

The combustion of fossil fuels from electricity generation or transportation is the major source for the emission of  $CO_2$  greenhouse gas. Calcium looping is one of the potential methods to capture  $CO_2$  [1–7]. Calcium oxide based material is used for  $CO_2$  capture because of its advantages such as high  $CO_2$  adsorption capacity, wide availability in natural minerals (e.g. limestone and dolomite), reversible carbonation/calcination reaction, and used

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http://dx.doi.org/10.1016/j.apsusc.2015.12.121 0169-4332/© 2015 Elsevier B.V. All rights reserved. adsorbents can be replaced as raw material in cement manufacturing [8,9]. Calcium hydroxide (Ca(OH)<sub>2</sub>) was investigated as one of the important candidates for high-temperature CO<sub>2</sub> capture [10–17]. However, the main drawback is the rapid decrease of adsorption performance during multiple cycles. This is mainly attributed to the sintering effect in each calcination step [18]. Therefore, the researchers have investigated on the synthesis of Ca(OH)<sub>2</sub> based adsorbents with high BET surface areas and mesopore size distribution to maintain the CO<sub>2</sub> adsorption capacity during multiple cycles [8,9,19–21]. The researchers have also focused on the system with low carbonation-decarbonation temperature for CO<sub>2</sub> capture [11,13,15,17,22–27]. The results are summarized in Table 1. The table clearly shows that most of the reactions were performed in the range of 700-800°C. Recently, Broda and Müller [28] studied the influence of calcination temperature on the CO<sub>2</sub> adsorption capacity CaO sorbents. They observed







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Table 1

Summary of calcium based adsorbents and their operating temperature for CO<sub>2</sub> capture.

Material	Synthesis method	Temperature (°C)		Reference
		Carbonation	Calcination	
Al <sub>2</sub> O <sub>3</sub> -CaO	Co-precipitation	750	750	[22]
SiO <sub>2</sub> -CaO	Mechanical mixing	800	800	[23]
Ca(OH) <sub>2</sub>	Commercial	620	805	[15]
Ca(OH) <sub>2</sub>	Commercial	620	850	[13]
CaO-MgO	Co-precipitation	700	700	[25]
CaO-MgO	Co-precipitation	750	750	[26]
ZrO <sub>2</sub> -CaO	Flame spray pyrolysis/physical mixing	700	700	[27]
Zr-CaO	Ultrasound	750	750	[28]
CaO	Sol-gel assisted hydrothermal	800	800	[17]
Zr-Ce/Ca(OH) <sub>2</sub>	Co-precipitation	800	800	[11]

that high calcination temperature ( $\geq 900 \,^{\circ}$ C) accelerated the thermal sintering of sorbent and reduced the CO<sub>2</sub> adsorption capacity during the cyclic use. Based on these findings, in this work the carbonation and calcination reactions were carried out at the same temperature (800  $^{\circ}$ C) in order to avoid the sintering of adsorbents between cyclic operations.

Cetyl trimethyl ammonium bromide (CTAB) was used as a surfactant to synthesize Ca(OH)<sub>2</sub> precursor at low temperature (40 °C) via precipitation method. Ca(OH)<sub>2</sub> sorbents prepared using CTAB showed high CO<sub>2</sub> adsorption performance when compared to other precursor adsorbents [11-16,27,29]. Recently, few researchers prepared the Ca-based adsorbents by sol-gel method using CTAB to develop the CO<sub>2</sub> adsorption performances [27,29]. Radfarnia and Iliuta [27] reported that Zr-stabilized CaO adsorbent prepared with CTAB possessed high CO<sub>2</sub> adsorption capacity during cyclic use. Akgsornpeak et al. [29] studied the CO<sub>2</sub> adsorption capacity of CaO adsorbents prepared via sol-gel method by varying Ca<sup>2+</sup>/CTAB molar ratios. Generally, the combustion of CTAB in the temperature range of 200-500 °C gives rise to the micro/mesoporous channels, which will enhance the diffusion of CO<sub>2</sub> during carbonation process. Therefore the use of CTAB for the synthesis of Ca(OH)<sub>2</sub> improves and maintains the CO<sub>2</sub> adsorption performance during cvclic use. The previous studies demonstrated that CO<sub>2</sub> adsorption capacity of CaO based sorbents was increased in the presence of bromide dopants [30–33].

In this present work, we reported the effect of CTAB concentration on the phase purity, crystallite size, morphology, surface area, thermal property,  $CO_2$  adsorption performance and cyclic stability of  $Ca(OH)_2$  precursor adsorbent. It was observed that the CTAB concentration played an important role in the  $CO_2$  adsorption performance of  $Ca(OH)_2$  based sorbents.

#### 2. Experimental

#### 2.1. Synthesis of Ca(OH)<sub>2</sub> precursor adsorbent

The Ca(OH)<sub>2</sub> precursor adsorbent was synthesized using cationic *n*-hexadecyltrimethyl ammoniumbromide (CTAB) (Kanto Chemical Co., Inc.), sodium hydroxide (NaOH) (Wako Chemical Co., Inc.) and calcium acetate monohydrate (Ca(CH<sub>3</sub>COO)<sub>2</sub>·H<sub>2</sub>O) (Wako Chemical Co., Inc.). All chemicals were of analytical grade and were used without further purification. Different concentrations of CTAB ranging from 0.2 M to 0.8 M were mixed with 1.6 M NaOH aqueous solution. Separately, this solution was heated to ~40 °C and vigorously stirred for 30 min. Then, 0.2 M of Ca(CH<sub>3</sub>COO)<sub>2</sub>·H<sub>2</sub>O aqueous solution was added dropwisely. The formed white precipitates were centrifuged, washed several times with distilled water followed by ethanol and then dried in an oven at 70 °C for 24 h [11]. The samples were denoted as CH-2, CH-4, CH-6 and CH-8. The commercial Ca(OH)<sub>2</sub> sample was labeled as CC.

#### 2.2. Characterization

The synthesized Ca(OH)<sub>2</sub> precursor adsorbent was characterized by the following techniques. The crystalline structures were characterized by X-ray diffraction (XRD) with a Rigaku X-Ray Diffractometer using Cu K $\alpha_1$  radiation ( $\lambda = 0.154056$  nm). The crystallite sizes were calculated using Scherrer equation  $(D = 0.9 \lambda / \beta \cos \theta)$ , where D is the crystallite size,  $\lambda$  is the X-ray wavelength,  $\beta$  is the full-width half-maximum and  $\theta$  is the diffraction angle. The functional groups were investigated by a JASCO FTIR-6100 FV Fourier transform infrared spectroscopy (FTIR) in the range of  $400-4000 \text{ cm}^{-1}$  with spectral resolution of  $4 \text{ cm}^{-1}$ . The surface morphologies were observed by a Carl Zeiss SUPRA 35 VP field emission scanning electron microscopy (FESEM). Thermal behavior was studied with the help of a Rigaku thermogravimetry analysis (TG–DTA) under N<sub>2</sub> gas flow. The Brunauer–Emmett–Teller (BET) surface areas were determined using three points N<sub>2</sub> adsorption isotherm (Quantachrome (Autosorb-1-MP) instrument).

#### 2.3. CO<sub>2</sub> adsorption measurement

The carbonation/calcination performance of Ca(OH)<sub>2</sub> precursor adsorbents were carried out in a TG–DTA apparatus with Thermoplus 2 software. In this study, 800 °C was selected to conduct both carbonation and calcination reactions to avoid the repetitive heating and cooling of the samples between cyclic operations. Initially, a small amount of sample (~6.5 mg) was placed in a platinum crucible and heated to 800 °C at a ramp rate of 10 °C/min under N<sub>2</sub> gas flow. After reaching 800 °C, N<sub>2</sub> gas was changed to 100% CO<sub>2</sub> gas to perform the carbonation process for 30 min. Then, the carbonated sample was calcined for 6 min under 100% N<sub>2</sub> gas flow. The carbonation and calcination processes were repeated at 800 °C for 10 cycles. The constant gas flow rate for CO<sub>2</sub> and N<sub>2</sub> was 40 ml/min. The carbonation conversion was calculated using the following equation;

$$CO_2 \text{ Conversion}, X_N(\%) = \frac{w_N - w_I}{w_I} \times \frac{M_{CaO}}{M_{CO_2}} \times 100\%$$
(1)

where  $X_N$  is the carbonation conversion,  $w_N$  is the weight (%) of the carbonated sample after N cycle(s),  $w_1$  is the initial weight (%) of the calcined sample,  $M_{CaO}$  and  $M_{CO_2}$  are molar masses of CaO and CO<sub>2</sub>, respectively.

#### 3. Results and discussion

#### 3.1. Characterization of Ca(OH)<sub>2</sub> precursor adsorbents

#### 3.1.1. XRD

The XRD patterns of  $Ca(OH)_2$  precursor adsorbents are shown in Fig. 1. All the diffraction peaks could be attributed to hexagonal phase  $Ca(OH)_2$  (calcium hydroxide), according to the standard Download English Version:

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