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ENHANCEMENT OF CALCIUM OXIDE (CaO) FOR CARBON DIOXIDE (CO₂) CAPTURE

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ABSTRACT

Calcium Oxide (CaO) has a potential to be developed as adsorbent material to capture CO_2 at high temperature. This work provides review on the performance of the sorbent in term of source of the material and also the methods of preparation. On the other hand, the experimental research on modification CaO using ethanol-water hydration method was performed. Result on the modified CaO using ethanol-water hydration method has show improvement on the capturing performance from 20.16% to 72.65% conversion in *thermo gravimetric analyzer* (TGA). In addition, the performance of this modified CaO has maintained over 50% conversion after 5 cycles. The *X-ray diffraction* (XRD) investigation concluded that the CaO was converted to the $Ca(OH)_2$ after modification and its characteristics have improved in which the surface area and pore volume has evolved from $2.18m^2/g$ and $0.006m^3/g$ to $29.22m^2/g$ and $0.157m^3/g$, respectively. The comparison with other similar work shows different in performance observed and this is due to different CaO source used. In conclusion, ethanol water hydration has proven enhancing CO_2 capture performance and it prolonged the performance of the adsorbent for multi-cycles utilization.
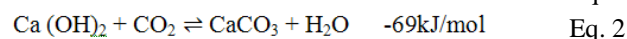
Keyword: CO_2 capture, adsorption, CaO-based, calcium oxide, calcium carbonate, ethanol-water hydration.

INTRODUCTION

The observed warming over the last 50 years is likely due to the increase of greenhouse gas emission such as carbon dioxide (CO_2), methane and nitrous oxide and CO_2 was identified as the main key factor(IPCC, 2007). Due to this awareness, many researches are performed with an aspiration to reduce CO_2 emission in the environment. Many techniques have been suggested in addressing the CO_2 emission issue such as (i) substitution to the green renewable feedstock and (ii) CO_2 capturing system at the exhaust of chemical processing system.

The application of renewable energy will contributes to "zero emission", but it is not widely available. Hence, controlling the CO_2 produced from a chemical plant by means of capturing is a favorable option. CO_2 could be capture using some adsorbent such as activated carbon and molecular sieve. However, these types of adsorbent are only active at low temperature and require high pressure. In example of gasification process, the effluent gases produced were usually in the form of hot gas stream (over 600°C). Hence, the gasses required cooling prior to capturing process. This is not favorable in term of energy efficient and cost effective (Metz *et al.*, 2005). As a solution for this, a material that can sustain the performance at high temperature shall be introduced. CaO-based sorbent has been identified as the suitable candidate for this job.

CO_2 capture using CaO is based on heterogeneous reactions, as shown in Eq. 1 and Eq. 2. The CO_2 adsorption using CaO is highly affected by the temperature and pressure of the system. Lin *et al*. (2004) addressed that CaO-based adsorbent is not well adsorb at low pressure in the coal gasification system. It is due to the equilibrium limit of the CO_2 partial pressure in the sorption material. High temperature above 800°C have tendency to reverse the reaction and release the CO_2. Numerous researches (Baker, 1962; Johnston, 1910; Smyth and Adams, 1923; Hill and Winter, 1956) have addressed the equilibrium or theoretical limit of the carbonation reaction of CaO which influenced by the temperature of the reaction and partial pressure of CO_2. And as the reaction involved is gas and solid reaction, the equilibrium constant is simplified as shown in the Eq. 3. Usually at atmospheric pressure, the maximum temperatures that can keep CO_2 concentrations below 1 vol% and 5 vol% (0.01 atm and 0.05 atm) are 642°C and 718°C, respectively (Monovic and Anthony, 2008b). Higher temperatures are unfavorable due to the reverse reaction.

$$CaO + CO_2 \rightleftharpoons CaCO_3 \qquad \text{-170.5kJ/mol} \qquad \text{Eq. 1}$$

$$Ca(OH)_2 + CO_2 \rightleftharpoons CaCO_3 + H_2O \qquad \text{-69kJ/mol} \qquad \text{Eq. 2}$$

$$K_{eq} = P_{CO_2}^{eq} = \exp\left(\frac{\Delta G_{rxn}^o(T)}{RT}\right) \qquad \text{Eq. 3}$$

As the capturing of the CO_2 using CaO-based material is

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reversible reaction based, it is practical to recycle the spent CaO. The release of CO_2, known as calcination, easily occurred at above 900^oC in atmosphere pressure. However, research on the multi-cycles calcination-carbonation shows that the performance of CaO-based sorbent was difficult to remained as high as the first cycle. High decrease in performance is observed after first cycle and it tends to stabilize by increasing the number of cycle. This similar findings trend has constantly reported by Florin and Harris (2008), Li *et al*. (2008a,b), Funnel *et al*. (2007) and Abanades (2002).

In recent years, many researchers worked on enhancing the performance especially for multi-cycles of the CaO through various modification methods such as hydration, acid treatment and chemical doping. The ethanol-water hydration method was observed the simplest and economically to apply. Li *et al*. (2008a) have discussed the performance of the prepared method. The effectiveness of this method especially on high conversion data in *fixed bed reactor* (FBR) has driven this study. Usually, most of the researchers performed the testing in the (TGA). In this micro amount sampler tester, the carbonation shall react efficiently compare to performance using larger reactor (in FBR). This work covered the performance testing using both TGA and FBR. In addition, this paper also reviewed on the enhancing CO_2 sorption in term of variety source and method of preparation or modification.

MATERIALS AND METHODS

Pure CaO was obtained from Sigma-Aldrich with powder size less than 100um and 99.9% absolute ethanol was obtained from Thermo-Fisher Brand. The modification was performed by mixing 70% ethanol concentration (35ml ethanol and 15ml distilled water) with CaO. They were mixed for 3hours before drying in the oven at 100^oC for 24hours. Both modified and non-modified samples were then subjected to the XRD and BET analysis and then to the carbonation test in the TGA and FBR.
The same sample was then tested in the TGA that continuously measured the weight gain when 60ml/min CO_2 is flowed in 140ml/min N_2. The carbonation condition is set at 650^oC for 15 minutes. The same sample also subjected to the fixed bed reactor (FBR) to perform similar carbonation reaction as in the TGA to imitate the work by Li *et al*. (2008a). The CaO conversion was calculated based on weight gain which describe in the Eq. 4.

$$Carbonation\ conversion, X = \frac{A, no.of\ mol\ of\ CaO\ reacted}{B, initial\ no.of\ mol\ of\ CaO} \times 100\% \quad \text{Eq. 4}$$

Where;

$$A = \frac{(W_f - W_i)}{MW_{CO_2}} \quad ; \quad B = \frac{W_i}{MW_{CaO}}$$

W_f – Final weight of the sample
W_i – Initial weight of the sample
MW_{CO2} – Molecular weight of CO_2
MW_{ads} – Molecular weight of adsorbent

RESULTS AND DISCUSSION

CaO-based sorption material is most abundantly available from natural resource such as limestone and dolomite. The sources of CaO are not limited to these two sources only. There are other researchers that proposed to use biomaterial sources such as from crab shell and oyster shell as the source of CaO (Jeon and Yeom, 2009; Barros *et al*., 2009; Nakatani *et al*., 2009).

The performance of the CaO based adsorbent varies depends on the source. The range of performance and surface area was wide. This is due to the different texture and pore size distribution of the CaO sorbent. Table 1 shows examples of the performance of different source of CaO, which are limestone and dolomite. It is shows that the performances for each source of limestone were not the same. Same goes to the dolomite.

Instead of the differences on the texture and pore size, the presence of impurities in the limestone is also beneficial in maintaining the treated sorbent's pore size and volume over multiple cycles (Hughes *et al*., 2004). The amount of impurities in the dolomite is much more compared to limestone. Hence, in comparison of limestone to dolomite performance, some researcher such as Silaban *et al*. (1995) and Grasa and Abanadez (2006) proved that the dolomite was better than limestone. Silaban *et al*. (1995) described that dolomite was superior to limestone as a sorbent especially for multi-cycle performance. It was due to differences between the structural properties of calcined dolomite and calcined limestone. The excess pore volume created by $MgCO_3$ decomposition is believed to be responsible for the more favorable cycling performance.

Recently, development on the material to capture CO_2 was focused in the area of developing method to enhance the capturing efficiency. The aims are either to improve the adsorption capacity and also the recyclability of the material in continuous process.

Gupta and Fan (2002) suggested that a mesoporous structure that maximizes the porosity would be less susceptible to pore pluggage, where it's provide sufficient surface area and ensure rapid kinetics. Due to that, they proposed modified precipitation technique which resulted in mesoporous structure with $60m^2/g$ surface area. The outcome of the modified precipitation technique shows that the performance of adsorbent maintained after 2 to 3 cycles.

Li *et al.* (2006) developed a new sorbent known as $CaO/Ca_{12}Al_{14}O_{33}$. The adsorbent was prepared by soaking the aluminium nitrate enneahydrate and CaO powder in propanol solution and followed some other steps described in their paper. In their explanation, $Ca_{12}Al_{14}O_{33}$ does not take part in the carbonation reaction. It was considered as an inert with a function as sintering retardant for CaO micro grain during calcination. The findings show that this type of adsorbent has achieved high CO_2 capture and cyclic stability throughout 50 cycles, where high conversion at least 50% was achieved.

Monovic and Anthony (2007, 2008b) investigated the steam reactivation of spent CaO based material. The application is not limited to improve capturing CO_2, it is also worth for capturing the SO_2. The tests have been performed on the spent limestone where the material was hydrated in vapor space which contained saturated steam. This method was similarly used by the Hughes *et al.* (2004) in their research. In this case, hydration by steam under pressure was considered as a method of reactivating sorbent spent in carbonation/calcination cycling. But it is not limited to spend CaO only, as this method can also be utilized to fresh adsorbent. The result found that capturing improvement is observed after the hydration. The hydrated sorbent have better properties than the spent sorbent where the increase in the surface area after reactivation method is observed.

Utilization of acetic acid solution for modification of dolomite was proposed by Li *et al.* (2008b). The acetic acid modification has changed the microstructure for dolomite which results in higher carbonation rate. The comparison made for modified and non-modified dolomite shows that the modification improves the conversion for multi-cycles adsorption. The modified adsorption exhibit the performance up to 94% conversion at $630^{\circ}C$ to $700^{\circ}C$. However, the drop in performance to 30% conversion was observed when the carbonation occurred at $750^{\circ}C$.

The same group doing acetic acid modification also investigated on the utilization of ethanol-water hydration technique for modification of limestone. In their research (Li *et al.*, 2008b), they found that the conversion for first cycle was increase to up to 10% compare to the non-modified sorbent. On the multi-cycles performance, the decay in performance of modified limestone was slower as opposed to original limestone. Ethanol was said as better anti sintering for the sorbent. The use of ethanol-water solutions during the hydration process decreased the mean particle and improved the porous structure of the calcined hydrate, mainly by increasing the porosity (Adanez, 1997).

Most recent development approach is toward utilizing the nano-technology. Lu *et al.* (2009) prepared nanostructure CaO by flammable spray pyrolysis. The successful of this method is due to creation of high surface area of sorbent in the range of $40-60m^2/g$. Due to small particle size, it gave advantage for rapid reaction controlled regime to take place. In term of stability performance over multiple cycles, it is shows that this prepared sorbent could maintained at 50% conversion after 40 cycles.

In order to promote long term multi-cycles adsorption, Florin and Harris (2009) testing the performance of nano-size $CaCO_3$ particle. For 20 minutes of carbonation reaction, they obtained a conversion up to 89% for the first cycle. However, the performance dropped to 21% after 100 cycles. The decay asymptote could be delayed by extending the duration of carbonation. The maximum conversion was achieved by them at 94% for the first cycle when the adsorbent is subjected to carbonation for 24hours.

Most of the methods described above have proven effective to improve the performance of the CaO especially for multi-cycles CO_2 capture. The selection of the methods is important and the criteria chosen should compensate the performance without ignoring the production cost.

Modification pure CaO through ethanol-water hydration method

The results of the study on the modification of CaO using ethanol-water hydration method are shown in figure1 to 4. Figure 1 show the result on the XRD analysis where Fig. 1(a) shows the result of non-modified CaO while figure 1 (b) shows result of the modified adsorbent. Based on the result, the non-modified compound consist trace of CaO and $Ca(OH)_2$. The existence of the $Ca(OH)_2$ was not expected as it is supposed to have only CaO compound. The existence of the $Ca(OH)_2$ was suspected due to the CaO has reacted with the moisture from environment during handling process for XRD analysis. The result after modification show that only trace of $Ca(OH)_2$ exist in the adsorbent. This has lead to the conclusion where the CaO has been converted to the $Ca(OH)_2$. The conversion to the $Ca(OH)_2$ is based on the Eq. 5.

$$CaO + H_2O \rightleftharpoons Ca(OH)_2 \qquad \text{Eq. 5}$$

In steam reforming gasification, Florin and Harris (2008) mentioned that the changed in the CaO to $Ca(OH)_2$ due to much higher concentration of H_2O steam in the system compared to the CO_2. This lead to assumption that the formation of $Ca(OH)_2$ creates the competition to the adsorption reaction in the gasification system. However, Lin *et al.* (2006) reported that improved of the adsorption CO_2 by $Ca(OH)_2$. This is due to $Ca(OH)_2$ is also reacted with the CO_2 to produce $CaCO_3$ and H_2O (Eq. 2). In this case, the $Ca(OH)_2$ has become the intermediate compound before adsorbing the CO_2.

Table 1. CO_2 capturing performance and surface area of variety source of CaO-based sorbent.

| Source	Type	Conversion of 1st cycle	Conversion at max study cycles	Performance test			Surface area (cm^2/g)	Other remarks	Ref.
				Pretreatment	Carbonation Condition	Calcination Condition			
Limestone	Kelly rock	~67.97-69.66%	10th cycle: ~34.26-39.52%	Calcined under nitrogen flow at 850 °C for 2 h.	Carbonated under 20%vol CO_2 flow at 650°C for 30min reaction	Calcined under 100%vol N_2 at 850°C for 30min reaction	4.06-4.89	Particle size: 75-150mm, 300-425mm, 600-750mm	Monovic and Anthony (2007, 2008a)
	Cadomin	~79%	10th cycle: ~40%	Not stated	Carbonated under 15%vol CO_2 flow at 650°C for 60min reaction	Calcined under 100%vol N_2 at 900°C for 60min reaction	14.92	sizes between 250 mm and 425 mm	Monovic and Anthony (2008a,b)
	Graymont	~79%	10th cycle: ~41%				7.06		
	Havelock	~86%	10th cycle: ~46%				14.44		
	Purbeck	n.a	n.a	Calcined under nitrogen flow at 750 °C for 500-600s.	Carbonated under 14%vol CO_2 flow at 750°C for 500s reaction	Calcined under 100%vol CO_2 at 750°C for 500s reaction	~24	Various particle size study ranging from 100-1180um	Fennel et al, 2007
	Penrith	n.a	n.a				~39	Size between 500-710um	
	Cadomin	n.a	n.a				~39		
	Havelock	n.a	n.a				~21.5		
	Glen Morrison	n.a	n.a				~33		
	Blanca	~68%	20th cycle: ~19%	Not stated	Carbonated under 0.1MPa CO_2 in air flow at 650°C for 5 min	Calcined under 0.1MPa CO_2 in air flow at temperature 850 °C for 5 min	n.a	Various particle size study ranging from 0.1-1mm	Grasa and Abanadez, (2006)
	Piasek	~70%	20th cycle: ~22%				n.a		
	Cadomin	~61%	20th cycle: ~16%				n.a		
	Havelock	~74%	20th cycle: ~20%				n.a		
	Gotland	~34%	20th cycle: ~10%				n.a		
	Imeco	~64%	40th cycle: ~12%		Carbonated under 0.1MPa CO_2 in air flow at 650°C for 5 min	Calcined under 0.1MPa CO_2 in air flow at temperature 850 °C for 5 min	n.a	300-600um	Grasa, Alonso and Abanadez, (2008)
	Piasek	~68%	50th cycle: ~11%				n.a		
Dolomite	Pilkington	~85%	2nd cycle: ~71%	Heated until 1000°C in N_2 and cooled in CO_2 flow until 800°C with a 30min dwell time twice.	Carbonated under 2L CO_2 flow at 800°C for 500s reaction	Calcined under 2L/min N_2 at 800°C for 500s reaction	n.a	165-180um	Galucci, Stendardo and Foscolo (2008)
	Sierra de arcos	~80%	20th cycle: ~40%	Not stated	Carbonated under 0.1MPa CO_2 in air flow at 650°C for 5 min	Calcined under 0.1MPa CO_2 in air flow at temperature 850°C for 5 min	n.a	Various particle size study ranging from 0.1-1mm	Grasa and Abanadez, (2006)
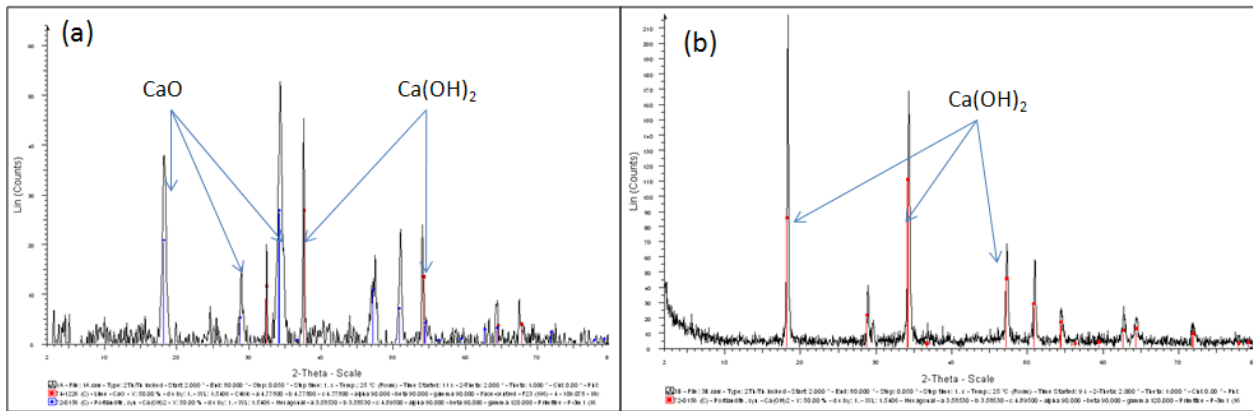
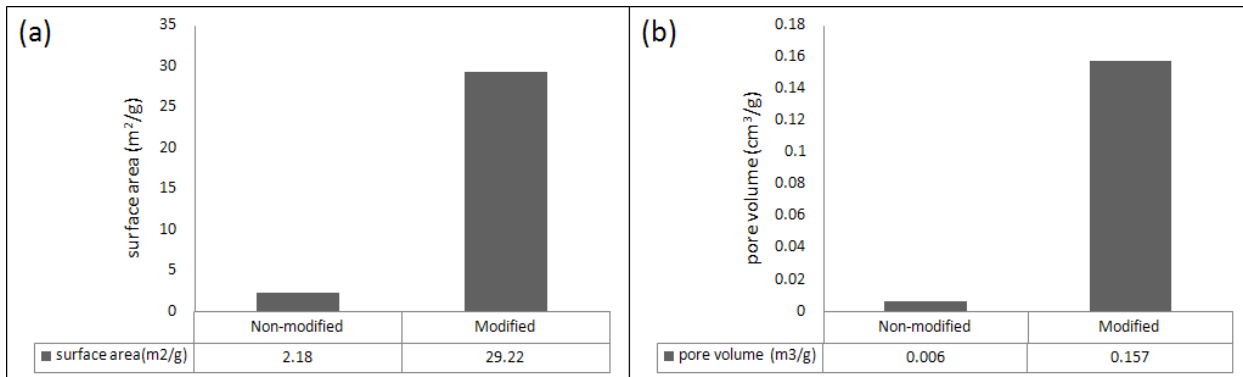Fig. 1. Result of XRD (a) non-modified (b) modified adsorbent.

Fig. 2. Result of (a) surface area and (b) pore volume.

The result on BET analysis is shown in figure 2, where figure 2 (a) is for the surface area and figure 2(b) is for the pore volume. It was found that the surface area and the pore volume increased after the modification from 2.18 to 29.22 m^2/g and 0.006 to 0.157cm^3/g respectively. The increased in surface area and pore volume will contribute to the high conversion of the adsorbent to capture CaO. This is proven by result on the carbonation conversion of the adsorbent in the figure 3. From figure 3, it is observed that the modification of the CaO successfully improved the carbonation conversion from 20.16% to 72.65% in TGA while 13.35% to 30.74% in FBR. The high pore size make the CO_2 easily pass through to the inner core of the CaO and promote high adsorption conversion by slowing the formation product layer thickness. Fast rate production product layer thickness will block the adsorbent pore and reduce the CO_2 intake in the inner core of the adsorbent. These structural limitations have prevented adsorbents from attaining theoretical conversions in the carbonation reaction. The use of ethanol was to stretch the pore structure of the CaO to larger surface area and pore volume. The result different between TGA and FBR was expected due to change in sample size from 5mg to 2g. Larger sample size is usually will be less efficient compare to the smallest sample.

Li *et al.* (2008[a]) reported the similar improvement using ethanol-water hydration to the limestone where improve in surface area and pore volume were observed from ~9 to ~14 m^2/g and ~0.08 to ~0.135cm^3/g, respectively. The improvement on both characteristics seem lower compared to the achievement in this work. This is due to the different source of the CaO used where pure CaO in this work obtained from the Sigma-Aldrich while they were using limestone in their research. This is another example on effect of the different source. As a result to this different source, the performance and characteristics of the prepared adsorbent will be varies.

The result on the multi-cycles performance is shown in figure 4 where the performance on the modified CaO remained higher than the fresh adsorbent even after five cycles and at least 50% conversion after five cycles. However, decrease in the performance was observed as the number of cycle increased. The decreased on the performance was due to the reduction of pore size and surface area during carbonation and calcination (Fennel *et al*., 2007). It was explained by sintering that is associated with the reduction of surface area in the material (Monovic and Anthony, 2007). The sintering effect was decreasing the available surface area by the increasing the product layer thickness ($CaCO_3$) on the reacting surface (Monovic and Anthony, 2008b).
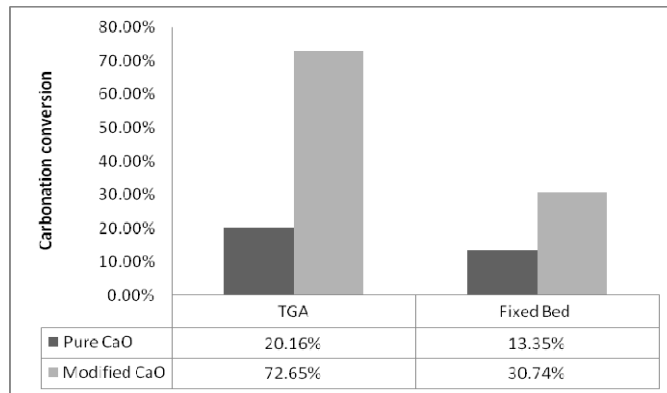
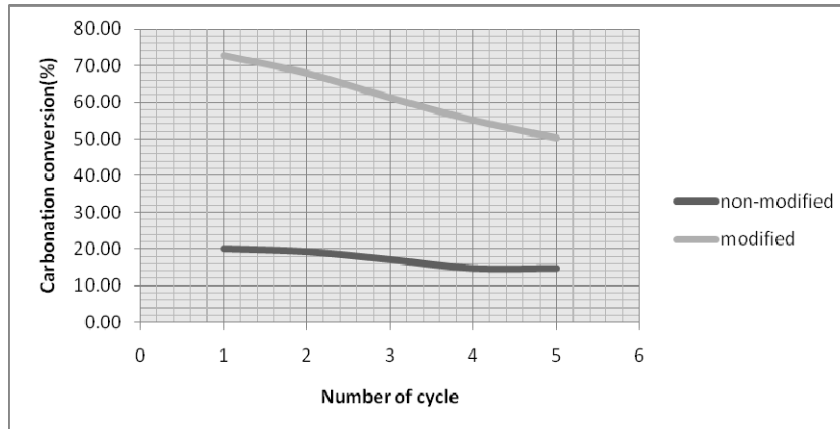Fig. 3. Carbonation conversion of pure CaO and modified CaO in TGA and Fixed Bed.

Fig. 4. Multi-cycles performance of modified and non-modified adsorbent.

CONCLUSION

Many researchers have proved their method successfully make the CO_2 capture better. In the present study, it is proved that the ethanol-water hydration successfully enhance CaO performance. However, the result was different from the other similar work which is expected due to different source of CaO used. In conclusion, different source and methods of preparation will contribute to different performance of adsorbent. Therefore, selection of source and method of improvement shall be emphasis to optimize the capturing of CO_2.

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