# CO<sub>2</sub> SELECTIVE CERAMIC MEMBRANE FOR WATER-GAS SHIFT REACTION WITH CONCOMITANT RECOVERY OF CO<sub>2</sub>

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## Abstract

Hydrotalcite material properties, specifically its  $CO_2$  reversibility, are critical to the performance of the proposed hydrotalcite-based membrane. In this report, we summarize the fundamental study we have performed using TGA, TGA/MS, and DRIFTS to quantify the degree of  $CO_2$  reversibility for the temperature range from 200 to 300°C. Results from these three separate studies consistently exhibit the  $CO_2$  reversibility. In addition, water effect appears negligible. Finally a high-pressure experimental study was performed to determine the reversibility under the actual operating condition. The results from this high-pressure ( $CO_2$ ) study also demonstrate the  $CO_2$  reversibility. In the next quarter, we will continue the high-pressure experiment in the presence of high-pressure steam to quantify its effect under the actual WGS environment. The quantitative information obtained from this study will then be incorporated in a mathematical model describing the  $CO_2$  permeance as a function of the membrane layer thickness.

# TABLE OF CONTENTS

1.	Introduction1
2.	Executive Summary1
3.	The Structure of the ALCOA # 2 Hydrotalcite1
4.	Investigation of the Effect of Heating Rate on Weight Loss2
	Experimental
5.	Reversibility Studies with the ALCOA #2 HTc
	Experimental
6.	Reversibility Studies in a High Pressure Flow Reactor with ALCOA #2 HTc12
	Experimental
7.	TGA Pressure Swing Adsorption Experiments with ALCOA #1 HTc14
	Experimental
8.	Conclusions
LIST	OF ACRONYMS AND ABBREVIATIONS

# List of Graphical Materials Figures

Page

Figure 1	XRD of Hydrotalcite sample, ALCOA #22
Figure 4	Cumulative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under inert atmosphere
Figure 5	Derivative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under inert atmosphere
Figure 6	Cumulative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under dry CO <sub>2</sub> atmosphere
Figure 7	Derivative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under dry CO <sub>2</sub> atmosphere
Figure 8	Cumulative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under humidified CO <sub>2</sub> atmosphere
Figure 9	Derivative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under humidified CO <sub>2</sub> atmosphere
Figure 10	CO <sub>2</sub> Reversibility study of hydrotalcite (ALCOA #2): Comparison of evacuation vs argon sweep during the desorption cycle
Figure 11	CO <sub>2</sub> Reversibility study of hydrotalcite (ALCOA #2): Comparison of adsorption cycle of 1, 2 and 3 hours
Figure 12	Weight gain during cyclic adsorption/desorption of hydrotalcite (ALCOA #2) under humidified CO <sub>2</sub> atmosphere and 1 hour adsorption
Figure 13	Weight loss during cyclic adsorption/desorption study of hydrotalcite (humidified CO <sub>2</sub> and 1 hour adsorption)
Figure 14	CO <sub>2</sub> and H <sub>2</sub> O weight gain of hydrotalcite (ALCOA #2) under humidified CO <sub>2</sub> and 1 hour adsorption (using TGA/MS.)
Figure 15	CO <sub>2</sub> weight gain/loss during cyclic adsorption/desorption study of hydrotalcite (ALCOA #2): measured by DRIFTS10
Figure 16	Water weight gain/loss during cyclic adsorption/desorption of hydrotalcite (ALCOA #2): Measured by DRIFTS11
Figure 17	Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 150°C
Figure 18	Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 150°C
Figure 19	H <sub>2</sub> O and CO <sub>2</sub> weight gain vs number of cycles (up to 14 cycles) during low pressure swing adsorption of hydrotalcite (AlCOA #1) at 150°C
Figure 20	Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 250°C
Figure 21	Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 250°C
Figure 22	H <sub>2</sub> O and CO <sub>2</sub> weight gain vs number of cycles (up to 14 cycles) during low pressure swing adsorption of hydrotalcite (AlCOA #1) at 2 50°C

# List of Graphical Materials Tables

Table 1	Weight loss vs temperature of hydrotalcite (ALCOA #2)2
Table 7	Weight gain during high pressure adsorption/desorption of hydrotalcite (ALCOA #2) at 250° C
Table 8	Weight gain during high pressure adsorption/desorption of hydrotalcite (ALCOA #2) at 200°C
Table 9	Weight gain during high pressure adsorption/desorption of hydrotalcite (ALCOA #2) at 150°C
Table 10	Operating condition for high pressure adsorption/desorption study of hydrotalcite (ALCOA #2)
Table 11	Weight gain during high pressure adsorption/desorption of hydrotalcite (ALCOA #2) using the condition described in Table 2

#### 1. Introduction

In one of our precious reports we presented results with the ALCOA # 1 sample, whose composition was  $Mg_{0.71}Al_{0.29}(OH)_2(CO_3)_{0.145}.0.46$  H<sub>2</sub>O. These results were also summarized in a publication [Yang *et al.*, Chem. Eng. Sci., 57, 2945, 2002]. We present result with a different Hydrotalcite which we call ALCOA # 2. First a series of experiments were carried out to establish the structure of the hydrotalcite itself. Subsequently we carried out a number of experiments to study the "reversibility" behavior of this hydrotalcite. At the end of this report we also present results with the original ALCOA #1 sample focusing again on issues of "reversibility" under a high pressure environment.

### 2. Executive Summary

In the previous report we have demonstrated the  $CO_2$  reversibility of hydrotalcite materials qualitatively. In this report we have determined the reversibility of  $CO_2$  in the range of 0.2 to 0.4 wt% under 1 atmosphere of  $CO_2$ . The degree of reversibility has been determined based upon the adsorption/desorption cyclic study for up to 14 cycles. This level of reversibility will be incorporated into the mathematical modeling we are developing to estimate the  $CO_2$  permeance of the membrane. In addition, the role of steam is very important for this membrane prepared for WGS reactions. Our results show that the effect of water on the reversibility of  $CO_2$  is negligible; however, no theoretical interpretation can be offered at this moment. Nevertheless, all the TGA, TGA/MS and DRIFTS study shows this level of reversibility in the presence and absence of steam. Water (or hydroxyl groups) after the first cycle shows no net gain in adsorption or no net loss in desorption; while  $CO_2$  shows reversible adsorption/desorption. More study will be performed in the next quarter to develop a hypothesis for the role of steam.

In addition to the surface study at 1 atmospheric pressure, a high-pressure experiment (i.e., 50 psi  $CO_2$ ) was performed in this quarter to determine the  $CO_2$  pressure effect on the degree of reversibility. Our preliminary result shows a similar level of reversibility as that obtained from the atmospheric study. In the next quarter, we will conduct the high pressure  $CO_2$  experiment in the presence of high-pressure steam to determine its effect on the degree of  $CO_2$  reversibility.

## 3. Structure of the ALCOA # 2 Hydrotalcite

#### **Experiment**

This HTc sample was provided by ALCOA and has been used as a model material for our study. In order first to identify whether the material is indeed an HTc we have carried out a XRD investigation. We then carried out an ICP and an TG/MS analysis to determine its composition.

## **Results/Discussions**

The material exhibits the typical HTc spectrum (see Figure 1) but the various peaks are nowhere as strong as the peaks that we observed with the original ALCOA # 1 sample. It is likely that the degree of crystallinity is less than the previous samples.

1



Figure 1 XRD of Hydrotalcite sample, ALCOA #2

Based on the results of this investigation the chemical formula of the ALCOA #2 sample is  $Mg_{0.645}Al_{0.355}(OH)_2(CO_3)_{0.178} \cdot 0.105(H_2O)$ . Table 1 below shows the weight loss observed by TG/MS and its corresponding result from to the ICP studies. The agreement is satisfactory. One note is that the ALCOA #2 sample, in addition to having a slightly different Al/Mg ratio, also contains significantly less interlayer water.

Weight Loss	1 <sup>st</sup> (H2O)	2 <sup>nd</sup> (OH from Al)	3 <sup>rd</sup> (OH from Mg)	4 <sup>th</sup> (CO2 from CO3)	Total Weight Loss
ICP Value	2%	11.0%	16.1%	10.9%	41%
Experimental from TG/MS	2%	12.4%	17.0%	9.6%	41%

 Table 1 Weight loss vs temperature of hydrotalcite (ALCOA #2)

## 4. Effect of Heating Rate on Weight Loss

One of the issues of concern about some of the observations and conclusions drawn with the study with the ALCOA # 1 sample pertained to the effect of heating rate. Are the effects observed of kinetic or equilibrium nature?

#### Experimental

It was clear for the ALCOA #1 sample that the observations made up to  $250^{\circ}$ C were of equilibrium nature, but some kinetic effects remained above this temperature. To investigate the same issues with the ALCOA #2 sample we studied the weight loss spectrum for 4 different heating rates namely 1 °C/min, 3 °C/min, 5 °C/min and 10°C/min. A fresh ALCOA #2 sample (110-120 mg) was used in every experiment, and

inert argon was utilized as a purge gas at a flow rate of 20 ml/min. The effect of varying the heating rate on weight loss was also studied in the presence of a dry and reactive atmosphere.

#### Results/Discussions

The experimental results for heating rate study under inert atmosphere are shown in Figures 4 and 5. For heating rates below 5  $^{\circ}$ C/min the weight loss curves are similar, particularly for temperatures below 430 $^{\circ}$ C (Figure 4). Clearly the results with the 10  $^{\circ}$ C/min heating rate contain kinetic effects, but for the lower heating rates the conclusions drawn are of an equilibrium nature.



Figure 4 Cumulative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under inert atmosphere.



Figure 5 Derivative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under inert atmosphere.

Under dry  $CO_2$  atmosphere, again, for all heating rates below 5°C/min, the TGA curves appears to coincide with one another (the dw/dt results show more scatter). Clearly the results for the heating rate of 10°C/min show considerable kinetic effects.



Figure 6 Cumulative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under dry CO<sub>2</sub> atmosphere.



Figure 7 Derivative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under dry CO<sub>2</sub> atmosphere.

The weight loss results in the presence of the humidified carbon dioxide atmosphere seem to indicate the absence of kinetic effects at all heating rates we have tried (See Figure 8 and 9).



Figure 8 Cumulative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under humidified CO<sub>2</sub> atmosphere.



Figure 9 Derivative weight loss vs temperature of hydrotalcite (ALCOA #2) with heating rate of 1, 3, 5 and 10°C/min and under humidified CO<sub>2</sub> atmosphere.

#### 5. Reversibility Studies with the ALCOA #2 HTc

In this section, the degree of  $CO_2$  reversibility at 1 atmospheric pressure was studied with TG, TGA/MS, and DRIFTS.

#### Experimental

Before the adsorption/desorption studies, we first investigated the effect of each key parameter involved in these experiments. For each series of experiments we utilized 100-120 mg of a fresh sample. During the adsorption part of the cycle, typically 30 cc/min of CO<sub>2</sub> was bubbled through a beaker containing distilled water. Measurements of the water concentration of the gas exiting the beaker indicated that the relative humidity of the  $CO_2$  stream after leaving the bubbler averaged 70%. The ALCOA #2 sample was exposed to this humidified CO<sub>2</sub> stream for varying periods of time. Subsequently, the flow of  $CO_2$  was shutdown and the desorption part of the cycle was initiated. We have investigated two different methods to carry out the desorption step. In the first method, upon termination of the  $CO_2$  flow, the sample was exposed to flowing Ar at a rate of 30 ml/min. Typically, after 30 min, the weight change of the sample ceased. We subsequently allowed the flow of Ar to continue for a total desorption period of 1 hr. In the second method, the TGA chamber was evacuated for a period of 1 hr at a pressure below 40 mTorr. For both methods, upon completion of the desorption step, the Ar flow is stopped (or the vacuum pump is shut down), and the sample is again exposed to humidified  $CO_2$  for 1 hr. During the second (and subsequent adsorption steps) the weight gain of the sample was again monitored. Typical results are shown in Figure 10. Notice that evacuation is a more effective means for carrying out desorption (~ a 10%) increase in weight gain). For the remainder of the study we utilized the evacuation procedure during the desorption step.



Figure 10 CO<sub>2</sub> Reversibility study of hydrotalcite (ALCOA #2): Comparison of evacuation vs argon sweep during the desorption cycle.

Subsequently we investigated the effect of varying the duration of the adsorption part of the cycle. The results are shown in Figure 11. Notice that increasing the adsorption step time from 1 to 2 hr increased the total amount adsorbed by about 5%. A subsequent increase from 2 to 3 hr exhibited no additional significant effect. For the remainder of the study we utilized, therefore, an adsorption step time of 3 hr. Upon completion of the preliminary runs, we studied the effect of temperature on the adsorption behavior of the ALCOA #2.

To validate the results obtained from adsorption/desorption using TGA/MS behavior further, we have also carried cyclic desorption/adsorption experiments *in situ* using DRIFTS. In our prior study we have also monitored the changes in an inert atmosphere of the DRIFTS peaks with temperature. In these experiments, starting from room temperature, the sample temperature was increased at a rate of  $0.5^{\circ}$ C/s. Every 20°C or so the temperature was kept constant, and the DRIFTS spectra were recorded after keeping the sample isothermal for a period of ~2 min. The testing was terminated when the temperature of the sample reached 580°C.

## **Results/Discussion**

The results of % weight gain via cyclic adsorption/desorption for the temperature ranging 150-350°C are shown in Figure 12. The % weight loss for the desorption cycle is presented in Figure 13. Figure 14 presents the  $CO_2$  and water weight gains via the MS analysis along with TGA. One can distinguish three different distinct regions of behavior. The first region is for temperatures below 190°C. In this region one observes that ALCOA #2 sample reversibly adsorbs only water and slight amounts of CO<sub>2</sub>. Previously we have reported experiments with the ALCOA#1 sample, in which the temperature of the same material was raised linearly in flowing Ar or in vacuum, and the thermal evolution of the hydrotalcite structure was monitored by a host of *in situ* surface techniques [Yang et al., Chem. Eng. Sci., 57, 2945, 2002]. The results of the cyclic adsorption/desorption experiments with the ALCOA #2 sample are consistent with the observations of Yang et al. [2002] with the ALCOA#1 sample. The cyclic adsorption/desorption experiments indicate that the exchange of interlayer water is a fairly reversible process (longer duration cyclic experiments with the ALCOA #2 sample are currently in process to validate the degree of reversibility - see below on the reversibility studies with the ALCOA #1 sample).



Figure 11 CO<sub>2</sub> Reversibility study of hydrotalcite (ALCOA #2): Comparison of adsorption cycle of 1, 2 and 3 hours.



Figure 12 Weight gain during cyclic adsorption/desorption of hydrotalcite (ALCOA #2) under humidified CO<sub>2</sub> atmosphere and 1 hour adsorption.



Figure 13 Weight loss during cyclic adsorption/desorption study of hydrotalcite (humidified CO<sub>2</sub> and 1 hour adsorption)



Figure 14 CO<sub>2</sub> and H<sub>2</sub>O weight gain of hydrotalcite (ALCOA #2) under humidified CO<sub>2</sub> and 1 hour adsorption (using TGA/MS.)

In the second region, 190 to  $280^{\circ}$ C, our prior studies with the ALCOA #1 sample under inert conditions indicate that the water that leaves is from hydroxyl groups that are bonded with Al cations. In addition, CO<sub>2</sub> is also emitted in this region. The current studies show that the same two species are also emitted during the desorption step of the first cycle of adsorption/desorption. However, only CO<sub>2</sub> appears to be reversibly adsorbed in this region. As can be seen in Figure 14, beyond the second desorption cycle very little  $H_2O$  is emitted, and the sample weight change can be fully attributed to the reversibly adsorbed  $CO_2$ .

In the third region,  $280 - 405^{\circ}$ C, our prior studies under inert conditions have indicated that the OH group bonded with Mg<sup>2+</sup> begins to disappear at 280°C and is completely lost at 405°C; a degradation of the hydrotalcite structure is also observed in the same region. Our cyclic adsorption/desorption experiments indicate that water and CO<sub>2</sub> are also emitted during the desorption step of the first cycle of adsorption/desorption. However, again, only CO<sub>2</sub> appears to be reversibly adsorbed in this region. As can be seen in Figure 14, the amount of CO<sub>2</sub> that is reversibly adsorbed in this region decreases as the temperature increases, consistent with our prior observations that the crystallinity of the hydrotalcite material decreases, and its structure begins to fall apart in this region.

The results from DRIFTS are shown in Figures 15 and 16. During these studies we have monitored a number of distinct peaks corresponding to various functional groups in the hydrotalcite. During our prior study [Yang et al, 2002] we have assigned the various peaks observed during DRIFTS studies of hydrotalcite as follows:

- (1) The DRIFTS signal at ~3470 cm<sup>-1</sup> is due to the OH group vibration in the Mg-Al-CO<sub>3</sub> hydrotalcite sample;
- (2) The DRIFTS signal at ~3070 cm<sup>-1</sup> is due to hydrogen bonding between water and the carbonate species in the interlayer space of the Mg-Al-CO<sub>3</sub> hydrotalcite sample;
- (3) The DRIFTS signal at ~1620 cm<sup>-1</sup> is due to the  $H_2O$  bending vibration of interlayer water in the Mg-Al-CO<sub>3</sub> hydrotlacite sample;
- (4) The DRIFTS signals at  $v_3=1370$  cm<sup>-1</sup>,  $v_2=940$  cm<sup>-1</sup>, and  $v_4=680$  cm<sup>-1</sup> at room temperature are due to the CO<sub>3</sub><sup>2-</sup> group vibration bands in the Mg-Al-CO<sub>3</sub> hydrotalcite sample



Figure 15 CO<sub>2</sub> weight gain/loss during cyclic adsorption/desorption study of hydrotalcite (ALCOA #2): measured by DRIFTS.

Weight changes based upon the DRIFTS measurement are summarized below:

- (1) The intensities of the interlayer water bands at 3070 cm<sup>-1</sup> and 1620 cm<sup>-1</sup> gradually decreases with increasing temperature, and disappear around 190°C.
- (2) The intensity of the OH vibration band at 3470cm<sup>-1</sup> begins to decrease at 190°C, and completely disappears at 440°C.
- (3) The band at 1370 cm<sup>-1</sup> for the  $CO_3^{2-} v_3$  vibration begins to decrease in size as the temperature increases, and also shifts to ~ 1350 cm<sup>-1</sup>. Gradually a band at 1530 cm<sup>-1</sup> begins to form at temperatures higher than 170°C. The band size at the lower wave number (~1350 cm<sup>-1</sup>) decreases as the temperature increases (and so are the peaks at 940 and 680 cm<sup>-1</sup>). At higher temperatures all peaks corresponding to  $CO_3^{2-}$  species in Mg-Al-CO<sub>3</sub> hydrotalcite disappear.



Figure 16 Water weight gain/loss during cyclic adsorption/desorption of hydrotalcite (ALCOA #2): Measured by DRIFTS.

Figure 15 shows the peak area reflecting the  $CO_3^{2-} v_3$  vibration during the cyclic adsorption/desorption experiments. Figure 16 shows the 3470 cm<sup>-1</sup> band corresponding to the OH<sup>-</sup> vibration and the changes for the integrated peak areas for the interlayer water peaks (3070 cm<sup>-1</sup> and 1620 cm<sup>-1</sup>). In these figures we show experimental data at the end of second and third adsorption and desorption steps for various temperatures. For temperatures below 190°C it is clear (consistent with the TGA/MS experiments) that the hydrotalcite during the cyclic sorption/desorption experiments simply exchanges reversibly interlayer water. Above this temperature, the interlayer water disappears and the hydrotalcite exchanges reversibly CO<sub>2</sub>. The amount of CO<sub>2</sub> that reversibly adsorbs decreases as a function of temperature. Clearly the hydrotalcite under the conditions of the cyclic experiments described here is not capable of reversibly exchanging the OH after a certain amount of OH desorbed during the first desorption cycle.

#### 6. Reversibility Studies in a High Pressure Flow Reactor with ALCOA #2 HTc

## Experiment

We have also studied the reversibility behavior of the ALCOA # 2 sample using a high-pressure adsorption flow system. Two mass flow controllers (one for argon, and the other for carbon dioxide) and one control valve was used to keep the system pressure constant during the experimental procedure. Before the cyclic reversibility experiments were carried out, a single adsorption experiment was performed in order to get some sense of flow rate changes resulting from the adsorption process. The system was first pressurized with dry argon to 50 psig, then temperature was increased to 250°C using a 5°C /min heating rate. And the system was kept at 250°C for 1 hr as a desorption step. When desorption step was over, the system was cooled down to 150°C in a flowing argon inert gas atmosphere. Subsequently the inlet gas was changed to CO<sub>2</sub> from argon while keeping the same pressure of 50 psig for 3 hrs as an adsorption step. During adsorption step, the outlet flow rate was monitored by a digital flow meter, and, from the flow rate change, the amount of adsorption was calculated.

We carried out a number of pressure swing experiments at various temperatures. In these experiments the sample was first heated to the desired temperature in hert gas, and the same temperature was kept constant during the whole experiment. Each cycle involved first evacuated the sample for 1 hr as a desorption step. After the evacuation step, the flow system was pressurized to 50 psig with argon. When the outlet flow rate was stabilized with argon at 50 psig, the inlet gas was then changed to  $CO_2$  for 3 hrs while maintaining the same pressure of 50 psig. Upon completing the first pressure swing cycle the procedure was repeated for a number of additional cycles and for a number of temperatures. The cyclic sorption experimental results for various temperature are summarized in Tables 8-10. In addition we performed a non-isothermal desorption, i.e., 250°C, for adsorption at 150°C. Table 10 is the summary of the experimental procedure followed for this adsorption experiment.

Table 2	Weight	gain	during	high	pressure	adsorption/desorption	of	hydrotalcite	(ALCOA	#2)	at
	250° C										

250°C	Weight gain at adsorption step (wt. %)
1 <sup>st</sup> Cycle	1.72
2 <sup>nd</sup> Cycle	1.69
3 <sup>rd</sup> Cycle	1.68
4 <sup>th</sup> Cycle	1.66

Table 3	Weight	gain	during	high	pressure	adsorption/desorption	of	hydrotalcite	(ALCOA	#2)	at
	200°C										

200°C	Weight gain at adsorption step (wt. %)
1 <sup>st</sup> Cycle	1.84
2 <sup>nd</sup> Cycle	1.79
3 <sup>rd</sup> Cycle	1.71
4 <sup>th</sup> Cycle	1.69

Table 4Weight gain during high pressure adsorption/desorption of hydrotalcite (ALCOA #2) at<br/>150°C

150°C	Weight gain at adsorption step (wt. %)

1 <sup>st</sup> Cycle	2.02
2 <sup>nd</sup> Cycle	1.97
3 <sup>rd</sup> Cycle	1.87
4 <sup>th</sup> Cycle	1.84

### Results/Discussion

The weight gains during adsorption are presented in Tables 7, 8, and 9 for the temperature, 250, 200 and  $150^{\circ}$ C respectively. In general the total weight gain is similar to those obtained from the low pressure experiment. However, the weight gain increases slightly along with the temperature increase for this temperature range, most likely resulted from the role of water at the lower temperature. In addition, the cyclic study here shows that the weight gain reaches a nearly steady state at the 4<sup>th</sup> cycle, instead of the 14<sup>th</sup> cycle from the low pressure study. No quantitative information for each individual species is available at this moment.

The weight gain obtained from the non-isothermal study is presented in Table 11 with the condition detailed in Table 10. The weight gain here is  $\sim$ 2.5wt%. much higher than  $\sim$ 11.8wt% from the isothermal study. Obviously the additional weight gain is most likely resulted from the additional desorption conducted at 250°C instead of 150°C.

	Step	Temperature (°C)	Pressure (psig)	Time	Gas
Desorption	Heating	r.t>250	50	~ 45 min (5 °C /min)	Ar only
	Isotherm	250	50	1 hr	Ar only
	Cooling	250 - → 150	50	20 min (-5°C/min)	Ar only
Adsorption	Isotherm	150	50	3 hr	CO2 only

 Table 5
 Operating condition for high pressure adsorption/desorption study of hydrotalcite (ALCOA #2)

The results from the non-isothermal study are summarized at the Table 12. The ALCOA #2 appears to show relatively reversible adsorption behavior under these conditions

Table 6	Weight	gain	during	high	pressure	adsorption/desorption	of	hydrotalcite	(ALCOA	#2)
	using th	e cond								

	Weight gain at adsorption step(wt. %)
1 <sup>st</sup> Cycle	2.621
2 <sup>nd</sup> Cycle	2.542
3 <sup>rd</sup> Cycle	2.476

7. TGA Pressure Swing Adsorption Experiments with >5 cycles.

This section focuses on the adsorption/desorption cyclic study up to 14 cycles to determine the ultimate reversible capacity under a atmosphere.

#### **Experiment**

We have also carried out pressure swing cyclic experiments with the ALCOA#1 sample. These experiments are of much longer duration that the cyclic experiments with the ALCOA #2 sample above. Two different temperatures were investigated 150  $^{\circ}$ C, and 250  $^{\circ}$ C. The amount of sample utilized was 10 mg, and the same sample was used for both experiments. For each experiment, the sample was first heated in UHP dry Ar (20 ml/min; 5  $^{\circ}$ C/min) and the weight of the sample and the gas composition was monitored. Upon reaching the desired temperature the feed was switched to humidified carbon dioxide (20 ml/min, 70% RH) and kept there for 3 hr. Then the sample is evacuated for 1 hr, and then switched back on to humidified carbon dioxide for 3 hr and so on. The experiment was terminated when the weight gain during adsorption and weight loss during desorption were unchanged in comparison with the weight lgain/loss obtained from the previous cycle.

#### Results/Discussions

Figures 17, and 18 show the weight change observed for a total of 14 cycles. Figure 19 shows the corresponding MS signals during the heating and evacuation parts of the cycle. Only water was detected coming out of the sample with mass spectrometer for the cyclic experiments at 150°C, an observation consistent with the results of the previously published paper (Yang et al., Chem. Eng. Sci., 57, 2945, 2002).



Figure 17 Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 150°C



Figure 18 Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 150°C



Figure 19 H<sub>2</sub>O and CO<sub>2</sub> weight gain vs number of cycles (up to 14 cycles) during low pressure swing adsorption of hydrotalcite (AlCOA #1) at 150°C

The experiments at 150°C indicate that the system reaches a steady state reversible behavior after the  $11^{th}$  cycle, with the corresponding weight change being 0.23%.



Figure 20 Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 250°C



Figure 21 Weight change vs number of cycles (up to 14 cycles) during low pressure swing adsorption study of hydrotalcite (ALCOA #1) at 250°C



Figure 22 H<sub>2</sub>O and CO<sub>2</sub> weight gain vs number of cycles (up to 14 cycles) during low pressure swing adsorption of hydrotalcite (AlCOA #1) at 2 50°C

Figures 20 and 21 show the weight change observed for a total of 14 cycles for the ALCOA #1 sample at 250°C. It is observed that ALCOA #1 sample reaches a steady state behavior after the 9<sup>th</sup> cycle, with the corresponding reversible weight change beinf 0.31%. Figures 22 shows the corresponding MS signals during the heating and evacuation parts of the cycle. During the heating part of the experiment both water and  $CO_2$  are emitted; however subsequently to that only  $CO_2$  is emitted indicating that under these low RH conditions the OH are not reversibly exchanged (a similar observation was also made for the ALCOA#2 sample during the shorter term reversibility experiments).

## 8. Conclusions

- 1) The heating rate study concludes that 5°C/min is sufficient to eliminate the kinetic effect in the TGA study with this type of hydrotalcite. For the rest of the experimental work, 5°C/min was chosen to obtain the equilibrium data. Thus, the properties obtained from this study represent the true thermodynamic properties with negligible effect from kinetics.
- 2) The TG. TGA/MS and DRIFTS studies (at 1 atmospheric pressure in the presence and absence of water) conclude that CO<sub>2</sub> can reversibly adsorb/desorb from hydrotalcite in the temperature range of 190 to 340°C. The amount of adsorption decreases from 2.0 to 1.7 wt% from the first to fifth cycles respectively. Water (or hydroxyl group) appears not involved in the CO<sub>2</sub> reversibility since no water adsorbed/desorbed after the first cycle.
- 3) The total weight gain ranges from 1.66 to 2.02 wt% for the temperature ranging from 150 to 250°C from the high pressure  $CO_2$  study. In the next quarter we will report the results from the MS analysis to determine the degree of  $CO_2$  reversibility under the high pressure  $CO_2$  environment. In addition the experiment in the presence of the high pressure steam will also be conducted.
- 4) The true degree of reversibility has been determined in the range of 0.3 wt% CO2 at 250°C based upon the cyclic adsorption/desorption study up to 14<sup>th</sup> cycles.

# List Of Acronyms and Abbreviations

HT:	Hydrotalcite
ICP:	Inductive Coupling Plasma
MS:	Mass Sepectroscopy
TGA:	Thermal Gravimetric Analysis
XRD:	X-ray Diffraction