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Adsorption of Carbon Dioxide on Chemically Modified High Surface Area Carbon-Based Adsorbents at High Temperature

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Abstract. The adsorption capacity of carbon dioxide on high surface area carbon-based adsorbents before and after chemical modification at 28°C and 300°C have been studied. The high adsorption capacity adsorbents for carbon dioxide at high temperature have been developed by introducing MgO and S—CaO—MgO on carbon-based adsorbents. Their adsorption capacities for carbon dioxide were 0.28 and 0.22 m mol/g at 300°C, 1 Bar, respectively.

Keywords: carbon dioxide, carbon-based adsorbent and high temperature

1. Introduction

Adsorption of gases on microporous adsorbents is receiving increased attention in view of the importance of removal and recovery of pollutants gases from air, especially those gases produced by various combustion processes. The large-quantity discharge of carbon dioxide into the atmosphere due to the consumption of large amounts of fossil fuels has become one of the most serious global environment problems, which is now required to be more seriously addressed worldwide, and has been widely discussed (Azar and Rodhe, 1997; Herzog et al., 1997; Ruether, 1999).

Several options exist to reduce these carbon dioxide emissions, including the substitution of nuclear power for fossil fuels, the substitution of natural gas for coal, separating and capturing the carbon dioxide prior to emission into the environment (Burchell and Judkins, 1996). All these techniques have the attractive feature of limiting the amount of carbon dioxide emitted into the atmosphere, but each has economic, technical or societal limitations. The removal and recovery of carbon dioxide from power plant fuel gases is considered to be one of the effective approaches for reducing the total carbon dioxide emissions from the energy point of view.

A number of techniques can be used for the separation of carbon dioxide from fuel gas streams. The large-scale separation of carbon dioxide by absorption is a commercial operation used throughout the world. Other techniques exist which could be considered for energy-related applications, such as, cryogenic separation, membrane separation and adsorption processes (pressure swing adsorption (PSA), vacuum swing adsorption (VSA) and temperature swing adsorption (TSA). In the last two decades, active research efforts have been placed on the separation of carbon dioxide by adsorption process and this technique has been industrialized. The key component of the adsorption process is the adsorbent. The development of synthetic and higher capacity, more selective adsorbent greatly improves the overall performance of the adsorption separation process. Adsorbents with large surface area such as activated carbons and zeolites, which have high adsorption capacity, high selectivity and rapid mass transfer in particle, have been widely investigated as the adsorbents for carbon dioxide separation. Activated carbon fibers and carbon fiber composite molecular

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sieves (CFCMS) recently emerged as promising materials (Burchell et al., 1997; Sircar et al., 1996).

Recently, Sircar et al., have been researching on sorption enhanced reaction process for production of hydrogen via steam methane reforming (SERP-SMR), and have made a great process (Carvill et al., 1996; Hufton et al., 1997a, 1998 and 1999; Sircar et al., 1995). The SERP concept combing chemical reaction and adsorption separation of reaction products in a single unit operation in conjunction with the adsorbent regeneration using the principles of PSA. In the SERP, the reforming reaction occurs at a significantly lower temperature (300–500°C) compared to the conventional steam methane reforming (800–1100°C) while achieving the same conversion of methane to hydrogen combined with higher purity H_2 (from 70–75% to 98%) directly from the SERP-reactor and significantly lower capital cost (from 1.0 to 0.69) (Hufton et al., 1998). However, the SERP is just like other adsorption separation processes, the first and most important issue is to find the appropriate adsorbent. In the SERP case this adsorbent must possess (1) high adsorption capacity for carbon dioxide at high temperature; therefore its adsorption capacity for carbon dioxide should be higher than 0.3 m mol/g at 300 $\sim$ 500°C in the presence of steam at high partial pressure and at low partial pressure of carbon dioxide (P_{CO2}, 0.1 to 1.0 atm; $P_{H2O}/P_{CO2} > 20$); (2) adequate adsorption/desorption kinetics for carbon dioxide at reaction and regeneration conditions; (3) stable adsorption capacity of carbon dioxide after repeated adsorption/desorption cycles; (4) adequate mechanical strength of adsorbent particles after cyclic exposure to high pressure steam (Hufton et al., 1997a, 1997b).

The adsorbent used in the SERP process (Hufton et al., 1999) is an hydrotalcite material with capacity of 0.52 mol/kg at 400°C, partial pressure of CO_2 of 0.7 bar under dry conditions. The adsorption capacity is higher in the presence of steam and a capacity decrease from 0.52 mol/kg to 0.45 mol/kg was observed after 60 cycles. These results are similar to those reported by Ding and Alpay (2000) for hydrotalcite adsorbent and better than those obtained by Zou et al. (2000) with commercial hydrotalcites.

Although there has been extensive research on the equilibrium of carbon dioxide on microporous adsorbents at ambient temperature and atmospheric pressure, for the purpose of elucidating pore structure and surface area of micropore adsorbents, and discussion of the heat of adsorption effect on the capacity of gas uptake (Cazorla et al., 1996; Frère et al., 1998; Groszek, 1997; Nakashima et al., 1995), there are only a few investigations relevant to adsorption of carbon dioxide at high temperature (Gaffney et al., 1999; Song and Lee, 1998).

This work is focused on developing a novel adsorbent for removal and concentration of carbon dioxide from process gas streams at elevated temperatures, and measurement of experimental single adsorption isotherms for carbon dioxide over commercial high surface area carbon-based adsorbents at 300°C.

2. Experimental

2.1. Materials and Reagents

2.1.1. Adsorbent. The adsorbent that was chosen in this study is a carbon-based adsorbent. It can be found from earlier studies that carbon-based adsorbent has high adsorption capacity for carbon dioxide (Kapoor and Yang, 1989; Reich et al., 1980; Yang, 1987; Zondlo and Sircar, 1978). The carbon dioxide adsorption on non-carbon-based sorbents, such as zeolites, is too strong, which makes desorption difficult. A further consideration on the selection of adsorbents concerns possible effect of the "impurities" in the hot streams, such as steam. Moreover, the strengths of adsorption of these impurities on non-carbon-based adsorbents are unfavorable for desorption. In this study, two types of commercial MAXSORB (MSC-30 and G08H) provided by Kansai Coke and Chemical Co. Ltd (Japan) were used. MAXSORB has high BET surface area and a large adsorption capacity in comparison with conventional activated carbons (Otowa et al., 1993). The physical properties of the two carbon-based adsorbents are presented in Table 1.

Table 1. The physical properties of MAXSORB (MSC-30[a] and GO8H[a]).

Property	MSC-30	GO8H
Form	Powder	Pellet
Surface area (m^2/g)	3370	2250
Total pore volume (ml/g)	1.8	1.1
Ash or (binder) (wt%)	0.2	20.4
Particle size	92 μm	0.8 mm

[a] Samples were from Kansai Coke and Chemical Co. Ltd, Japan.

2.1.2. *Reagents.* Introduction of alkali metal or alkaline earth on the surface of the adsorbent provides basic sites which have strong affinity for molecules of acidic gases like carbon dioxide. The basicity of a metal oxide decreases when the ratio of electric charges to the radius of the metal ion increases (Auroux and Gervasini, 1990). Calcium oxide and magnesium oxide have low values of this ratio, and therefore they can provide strong basic sites on the surface of the adsorbent. Furthermore, it was reported that calcium oxide and magnesium oxide impregnated to inorganic memberanes can enhance the adsorption of carbon dioxide at high temperature (Cho et al., 1995; Uhlhorn et al., 1989). Calcium acetate monohydrate and magnesium acetate tetrahydrate were chosen in the study as calcium oxide and magnesium oxide precursors, respectively, because they are easily dissolved in water and the products after calcination have large pore volume. The carbon dioxide is 99.998% purity provided by Air Liquide (France). Calcium acetate monohydrate, magnesium acetate tetrahydrate and sodium dodecyl sulfate are 99+%, 99% and 99% purity, respectively, from Aldrich Company.

2.1.3. *The Chemical Modification Process Description.* An amount of chemical reagent(s) was (were) dissolved in deionized water for 5 hr with magnetic stirring, then the solution was filtered to remove undissolved salt, and one gram of MSC-30 was impregnated with filtered solution for 1hr at room temperature, filtered and dried for 10 hr at 120°C and, finally, the impregnated MSC-30 was calcined at 700°C for 2 hr under nitrogen atmosphere. The technological scheme of chemically modification process is shown in Fig. 1, and reagent(s) and amounts used are listed in Table 2.

2.2. *Apparatus and Procedure*

2.2.1. *Apparatus.* A schematic diagram of the experimental apparatus for the single adsorption isotherms is showed in Fig. 2. It has three major sections: weighting system, gas providing system, and data acquisition. weighting measurements were performed with a microblance (A) (CI-Robal, Wilshire, UK) in which a cage with samples inside is suspended in one of his arms (B).

2.2.2. *Procedure.* Experiments for the single adsorption isotherms were carried out as follows. A small amount of carbon-based adsorbent sample (40–60 mg) was introduced in the microbalance basket, and the sample was submitted to a controlled temperature ramp of 4 ∼ 5 K/min in vacuum conditions (total pressure of ∼2 mbar) until the required temperature was reached. The sample was kept at the required temperature and ∼2 mbar for 3 ∼ 4 hr until no further variations in the weight were detected. The required temperature of the single adsorption isotherms is fixed in the oven, and the system is kept waiting until steady conditions are achieved. The experimental isotherm starts from the vacuum condition of 2 mbar (adsorption path), then introducing small amounts of the carbon dioxide step by step until the atmospheric pressure is reached.

3. Results and Discussion

3.1. *Effect of Adsorbent on Carbon Dioxide Adsorption Equilibrium*

Adsorption equilibrium isotherms of carbon dioxide on MSC-30 and G08H at two different temperatures are

Table 2. The reagents and amounts used in the chemical modification process.

Reagent and amount	Deionized water (ml)	Filtered solution (ml)	Sample name
10.2 g$(CH_3COO)_2Ca \bullet H_2O$	20	12	No. 5
12.5 g$(CH_3COO)_2Mg \bullet 4H_2O$	25	15	No. 6
5 g$(CH_3COO)_2Mg \bullet 4H_2O$	20	13	No. 7
5 g$(CH_3COO)_2Ca \bullet H_2O$			
5 g$(CH_3COO)_2Mg \bullet 4H_2O$	20	13	No. 8
5 g$(CH_3COO)_2Ca \bullet H_2O$			
0.4 g$CH_3(CH_{12})_{11}OSO_2Na$			
2.5 g$(CH_3COO)_2Mg \bullet 4H_2O$	30	24	No. 9
2.5 g$(CH_3COO)_2Ca \bullet 4H_2O$			

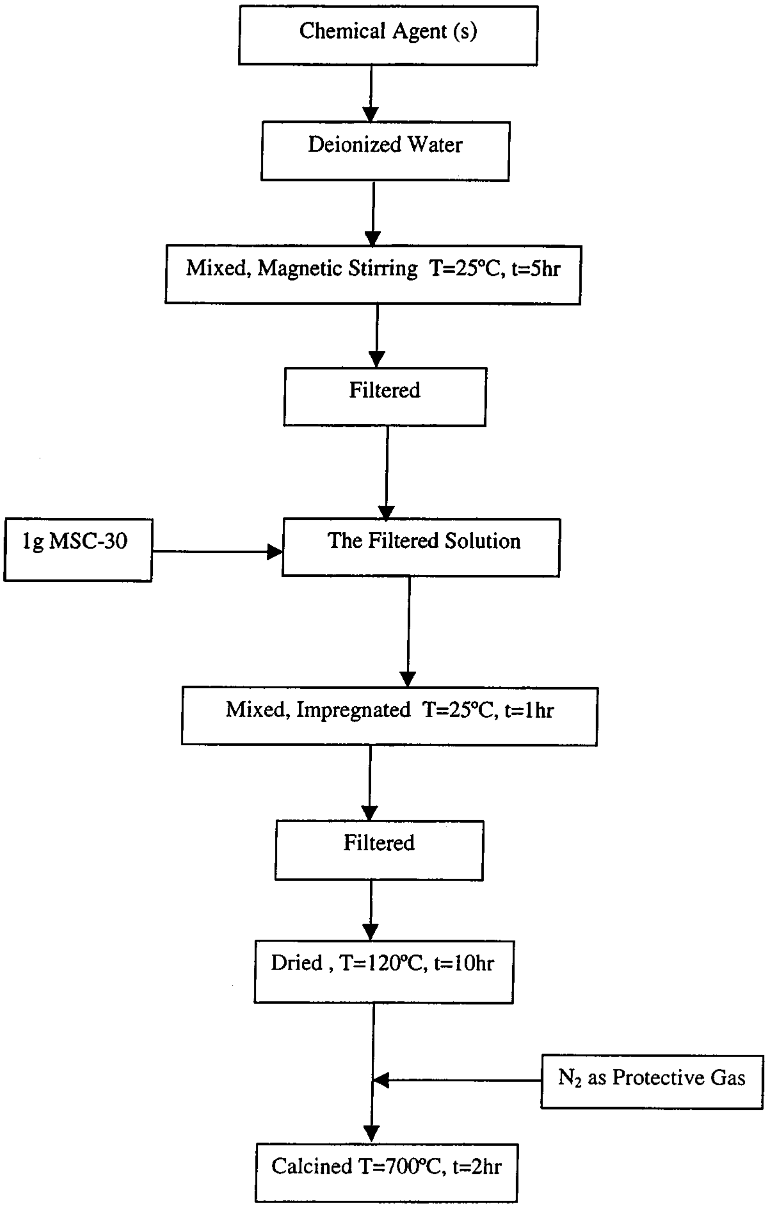

Figure 1. Technological scheme for chemical modification of MSC-30.

shown in Fig. 3. The results showed that (1) the amounts of adsorbed carbon dioxide on the two carbon-based adsorbents decreased with increasing temperature; (2) the difference of the adsorption capacity on the MSC-30 and G08H at 300°C is very small, which means that the effect of the microstructure (such as the surface area) of carbon-based adsorbents on adsorption capacity for carbon dioxide is becoming lower when the temperature is increasing. (3) the adsorption capacity of carbon dioxide on the two carbon-based adsorbents at 300°C and approximately 1 Bar are all very small (0.17 and 0.13 m mol/g, respectively). The results showed that the adsorption of carbon dioxide on carbon-based adsorbents is more difficult at this temperature without chemical modification. Similar results were reported previously (Song and Lee, 1998).

Generally, the amount of physically adsorbed gas is proportional to the partial pressure at low relative

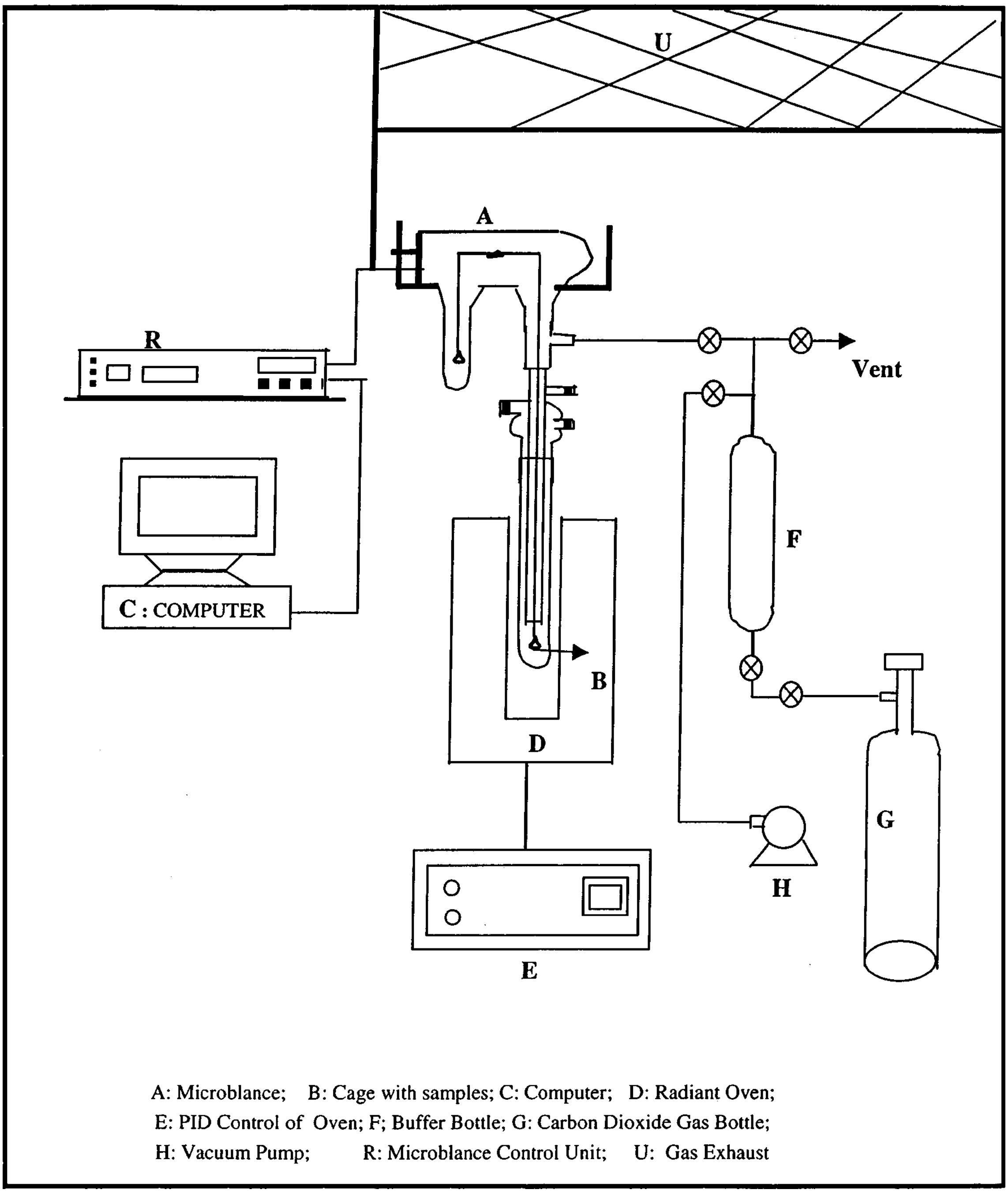

Figure 2. Schematic diagram of an experimental apparatus for measuring adsorption equilibrium isotherm.

pressure. The adsorption isotherms follow Henry's law in this case. Therefore the adsorptivity of the two adsorbents can be represented by Henry's constant. Table 3 shows Henry's constants of the two adsorbents at two different temperatures. Table 3 also shows that (1) the Henry's constants on the two carbon-based adsorbents decreased with increasing temperature, (2) the difference of the Henry's constants on the MSC-30 and

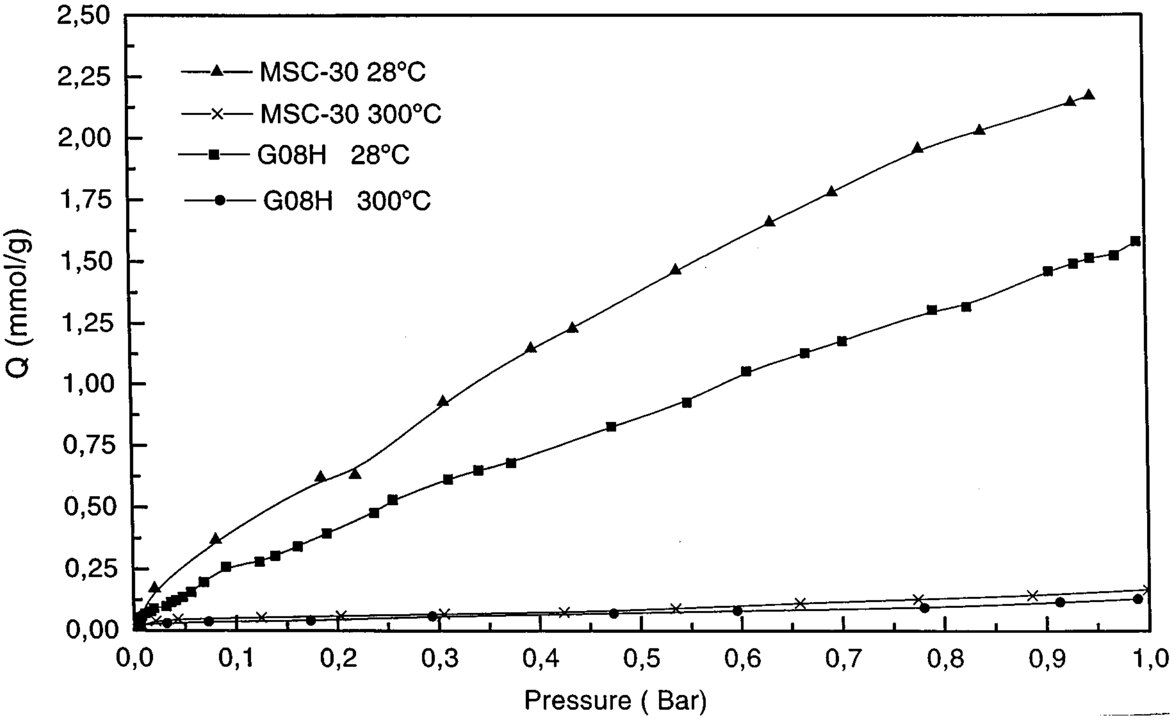

Figure 3. Adsorption of carbon dioxide on MSC-30 and G08H at 28 and 300°C.

G08H at 300°C is very small. However, it should be noted that the reversibility of the isotherms, the effect of moisture and the kinetics of sorption was not tested yet.

From the above discussions, the adsorption capacity of carbon dioxide on MSC-30 is higher than the one for G08H. Therefore, in this study, MSC-30 was chosen as the orginal adsorbent to be chemically modified.

3.2. *The Effect of Chemical Modification on Adsorption Capacity of Carbon Dioxide*

Figures 4 and 5 show the results of adsorption capacity of carbon dioxide on MSC-30 before and after chemical modification at 28°C and 300°C, respectively. The results show that the amounts of adsorbed carbon dioxide on the chemically modified MSC-30 adsorbents decreased with increasing temperature. The effect of chemical modification on the adsorption capacity of carbon dioxide is explained in the following sections.

Table 4 shows the BET surface areas of the various carbon-based adsorbents (samples 5 to 9) used in this work. It can be seen that the BET surface area of each chemically modified carbon sample is lower than that of the original carbon.

3.2.1. Single Metal Oxide. It was reported that calcium acetate monohydrate and magnesium acetate

Table 3. Henry constant for carbon dioxide on MSC-30 and G08H.

	Henry's Constant (m mol/g.Bar)	
Sample	28.1°C	300°C
MSC-30	2.27	0.14
G08H	1.54	0.11

Table 4. BET surface area for Carbon-based adsorbents.

Sample	Surface area (m^2/g)
5 (CaO-doped-MSC-30)	1366
6 (MgO-doped-MSC-30)	1272
7 (CaO—MgO-doped-MSC-30)	1357
8 (S—CaO—MgO-doped-MSC-30)	1545
9 (dil.CaO-MgO-doped-MSC-30)	1635

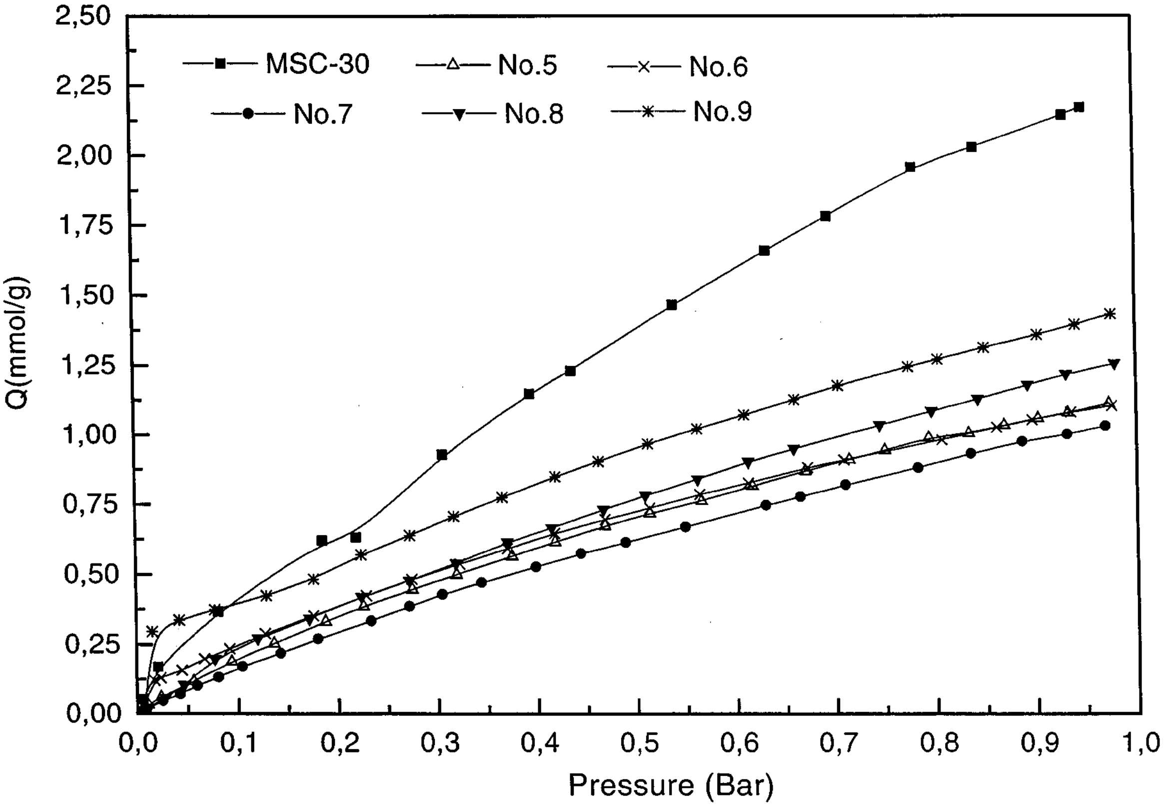

Figure 4. Adsorption of carbon dioxide on MSC-30 before and after chemical modification at 28°C. Sample 5—CaO-doped-MSC-30; Sample 6—MgO-doped-MSC-30; Sample 7—CaO—MgO-doped-MSC-30; Sample 8—S-CaO—MgO-doped-MSC-30; Sample 9—diluted CaO—MgO-doped-MSC-30.

tetrahydrate were converted into calcium oxide and magnesium oxide through the following pathways during calcination (Song and Lee, 1998).

$$(CH_3COO)_2Ca \bullet H_2O \longrightarrow (CH_3COO)_2Ca$$

$$\longrightarrow CaCO_3 \longrightarrow CaO$$

$$(CH_3COO)_2Mg \bullet 4H_2O \longrightarrow (CH_3COO)_2Mg$$

$$\longrightarrow MgCO_3 \longrightarrow MgO$$

It can be seen from Fig. 4 that adsorption capacities of CaO-doped MSC-30 and MgO-doped MSC-30 at 28°C are identical, and smaller than the one for MSC-30. However, Fig. 5 shows that the adsorption capacities of CaO-doped MSC-30 and MgO-doped MSC-30 at 300°C are very different. The adsorption capacity of MgO-doped MSC-30 (sample 6) was much higher than the one for MSC-30 (70%), but the adsorption capacity of CaO-doped MSC-30 (sample 5) is a slightly higher than that of MSC-30 at $P < \sim 0.9$ Bar, which is not in agreement with results reported by Song (Song and Lee, 1998). The reason is that the amount of adsorption on an adsorbent depends on the available surface area and the affinity for the adsorbates (Gregg and Sing, 1982). At 300°C the adsorption capacity of Sample 6 (MgO-doped MSC-30) is much higher (70%) than that of sample 5 (CaO-doped MSC-30), although BET Area of sample 5 (1366 m^2/g) is slightly higher than that of sample 6 (1272 m^2/g). This fact proves that the process is controlled not only from physisorption.

Chemical modification has two functions. One is to introduce metal oxides, and provide more strong basic sites which induces a stronger affinity for carbon dioxide on carbon-based adsorbent surface. As an example: carbon dioxide on calcium oxide will form the unidentate species, and carbon dioxide on magnesium oxide will be bidentate and unidentate species (Auroux and Gervasini, 1990). The other leads to the decrease in pore volume and available surface area for all adsorbents, particularly in this study, since the MSC-30 is a

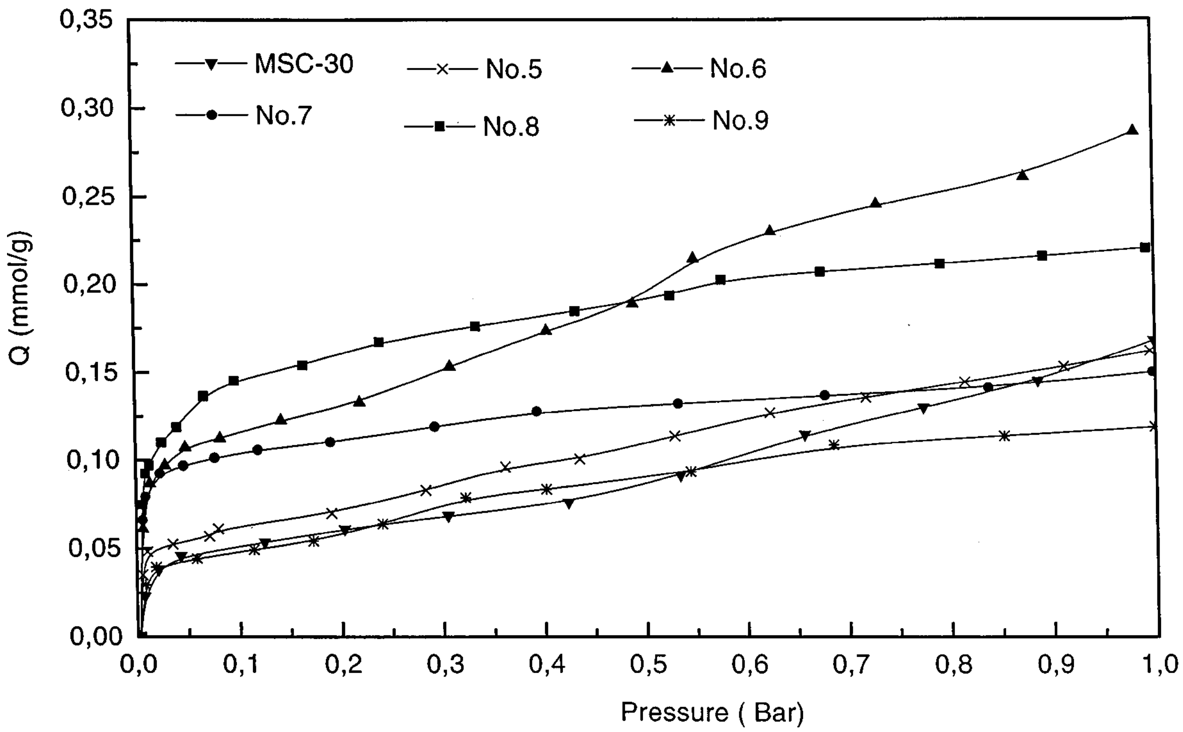

Figure 5. Adsorption of Carbon Dioxide on MSC-30 before and after chemical modification at 300°C. Sample 5—CaO-doped-MSC-30; Sample 6—MgO-doped-MSC-30; Sample 7—CaO—MgO-doped-MSC-30; Sample 8—S-CaO—MgO-doped-MSC-30; Sample 9—diluted CaO—MgO-doped-MSC-30.

high microporous carbon-based adsorbent. The former can enhance adsorption of carbon dioxide, but the latter is very unfavorable for adsorption of carbon dioxide. Furthermore, magnesium oxide has high surface area and high adsorption capacity of carbon dioxide, such as $S_{\mathrm{MgO}} = 180\,\mathrm{m}^2/\mathrm{g}$, $Q_{\mathrm{CO2}} = \sim 0.44\mathrm{m\,mol/g}$ at 300°C, 1Bar. (Sircar et al., 1995).

3.2.2. Double Metal Oxide. It can be seen from Fig. 4 that adsorption capacities of three CaO-MgO-doped MSC-30 samples at 28°C were all smaller than that of MSC-30, except for No. 9 at very low pressure ($<$0.1 Bar). At 28°C, the BET Surface Area of Sample 7 (1366 m^2/g) is lower than that of Sample 9 (1635 m^2/g), since the impregnation of carbon-based adsorbents with more concentrated alkali metals leads to the decrease in pore volume and available surface area and the adsorption capacity of Sample 7 is also lower than that of Sample 9. Figure 5 shows that a small amount of calcium oxide-magnesium oxide mixture introduced on the carbon-based adsorbent does not enhance adsorption of carbon dioxide at 300°C, but will decrease adsorption capacity of carbon dioxide on a carbon-based adsorbent; even if the calcium oxide-magnesium oxide mixture amount introduced is higher, its adsorption capacity was higher than that of MSC-30 at 300°C, only at $P < \sim 0.7$ Bar, because the positive effect decreases when the pressure increases. At 300°C, the adsorption capacity of Sample 7 is higher than that of sample 9 although Sample 9 has a higher surface area than Sample 7. This can be explained by the fact that Sample 7 has higher concentration of alkali metal oxides, that play a role in the adsorption at high temperature.

Effect of Surfactant. By adding a very small amount of surfactant (such as: $\mathrm{CH_3(CH_{12})_{11}OSO_2N_a}$), the adsorption capacity of CaO-MgO-doped MSC-30 at 300°C is greatly enhanced. Compared with MSC-30, the adsorption capacity of S—CaO—MgO-doped MSC-30 at 300°C is about 1.3 times that of MSC-30; compared to CaO—MgO-doped MSC-30, the adsorption capacity of S—CaO—MgO-doped MSC-30 at 300°C is about 1.47 times that of CaO—MgO-doped

MSC-30. The increase in capacity when the surfactant is present is due in part to the increase in the surface area of Sample 8 (1545 m^2/g), when comparing with Sample 7 (1357 m^2/g). Moreover, the carbon dioxide is a quadropole and has a polarizability; the heteroelement "S" is introduced on the carbon-based adsorbent, creates some new "activate species", and increases polarity of carbon-based adsorbent which results in stronger attraction of carbon dioxide on the surface of micropores and/or macropores within carbon-based adsorbents, thus increasing the adsorption capacity of carbon dioxide. The results indicate that (a) it is very difficult to enhance the adsorption capacity of carbon dioxide on carbon-based adsorbent at high temperature (300°C), and relative high pressure (1 Bar) by introducing calcium oxide-magnesium oxide mixture only. (b) introduction of a heteroelement "S" and increasing the polarity of carbon-based adsorbent is very favorable for the adsorption of carbon dioxide on carbon-based adsorbent at high temperature.

4. Conclusion

The adsorption capacity of carbon dioxide on a carbon-based adsorbent at high temperature can be greatly enhanced by simple chemical treatments, which introduced metal oxides and a heteroelement. The chemically modified carbon-based adsorbent can be directly used for the removal and recovery of carbon dioxide from power plant fuel gases. It is also very promising to be used in Sorption Enhanced Reaction Process when its adsorption capacity is improved.

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