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## Adsorption of carbon dioxide by sodium hydroxide-modified granular coconut shell activated carbon in a fixed bed

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#### **ABSTRACT**

In the present work, commercial coconut shell activated carbon was impregnated with alkaline NaOH to investigate the efficiency of modified activated carbon for  $CO<sub>2</sub>$  adsorption in a fixed-bed column adsorption system. The modification parameters, such as the NaOH concentration  $(24-48%)$  and dwelling time  $(1-4 h)$ , were also investigated. The results showed that a 32% NaOH concentration with a 3 h dwelling time provided the best CO2 adsorption capacity. Later, the modified activated carbon was characterized by nitrogen adsorption-desorption, scanning electron microscopy and Fourier transform infrared spectroscopy. The effects of the  $CO<sub>2</sub>$ % in the feed, the adsorption temperature, the feed flow rate and the amount of adsorbent in the column were investigated in the adsorption experiments. The maximum CO<sub>2</sub> adsorption capacity in this study was 27.10 mg/g at 35 °C. This study also suggests that NaOH-modified activated carbon is a state-of-the-art adsorbent for  $CO<sub>2</sub>$  adsorption.

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#### 1. Introduction

GHGs (global warming and greenhouse gases) are highly discussed topics around the world. Of all the GHGs,  $CO<sub>2</sub>$  has been regarded as a major contributor to the present global warming trend and is mainly emitted from anthropogenic sources. Thus, global awareness about  $CO<sub>2</sub>$  emissions has increased during the last decades, leading to increasing efforts to reduce their environmental impact, including preventive and remediation methods. To combat  $CO<sub>2</sub>$ , innovative  $CO<sub>2</sub>$  capturing technologies are needed. Several techniques have been proposed to capture  $CO<sub>2</sub>$ , including chemical absorption, physical adsorption and membrane separation  $[1–5]$  $[1–5]$ . Of these techniques, chemical absorption with an aqueous solution of alkanolamines, such as monoethanolamine and diethanolamine, is the most popular  $[6-8]$  $[6-8]$ . However, there are some problems associated with this technique, such as high corrosion, oxidative degradation of absorbents, and foaming in the gas-liquid interface [\[9\]](#page--1-0). Adsorption is a widely used technology for gas treatment due to its versatility and efficiency. For this method, solid porous adsorbents, such as activated carbon, can act as suitable alternatives for capturing  $CO<sub>2</sub>$  because activated carbons are insensitive to

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moisture, have large surface areas and have distinct porosities, ultimately leading to high adsorption capacities. Moreover, the regeneration of ACs (activated carbons) is possible at a low cost.

The surface chemistry of the activated carbon can be modified by chemical activation to increase the adsorption capacity. Recently, several excellent research initiatives have been taken to removal of  $CO<sub>2</sub>$  by modified activated carbon as an adsorbent. Caglayan and Aksoylu [\[10\]](#page--1-0) observed the substantial adsorption capacity of  $CO<sub>2</sub>$  by commercial activated carbon through modified with  $HNO<sub>3</sub>$  oxidation, air oxidation, alkali impregnation and heat treatment under helium gas atmosphere. In another study, by Yin and other coauthors  $[11]$  reported that the effect of  $N_2$  has little influence of the  $CO<sub>2</sub>$  adsorption on activated carbon. Three types of sterically hindered amines were successfully impregnated onto the surface of palm shell-based activated carbon and found higher adsorption capacity than virgin activated carbon [\[12\]](#page--1-0). Activated carbon was modified by washing with acid mixture of concentrated  $HNO<sub>3</sub>$  and  $H<sub>2</sub>SO<sub>4</sub>$ , and it was found that the adsorption capacities of the modified samples were increased upto 70% [\[13\]](#page--1-0). In one study by Shafeeyan et al. [\[14\]](#page--1-0) reported that the commercial GAC (granular activated carbon) became an efficient adsorbent for  $CO<sub>2</sub>$  when modified by using thermal treatment with ammonia. But activated carbon modified with NaOH for  $CO<sub>2</sub>$  adsorption is scarce in the literature. Because  $CO<sub>2</sub>$  is an acidic gas, treatment with an alkaline compound can provide more active functional sites for  $CO<sub>2</sub>$ .

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Activation with sodium hydroxide (NaOH) is more effective in the preparation of activated porous carbon because NaOH is inexpensive, minimally corrosive and environmentally friendly [\[15,16\]](#page--1-0).

In the present work, granular coconut shell activated carbon was modified with NaOH for  $CO<sub>2</sub>$  adsorption. The effects of different NaOH concentrations, contact times, gas flow rates, temperatures, and  $CO<sub>2</sub>$  concentrations on the adsorption process were also studied in a fixed-bed adsorption mode.

#### 2. Materials and methods

#### 2.1. Materials

Coconut shell-based activated carbon was purchased from Laju Carbon Products Sdn. Bhd., Malaysia. Granular activated carbons with particle sizes ranging from 0.85 to 0.425 mm were used in this study. Sodium hydroxide (NaOH) was purchased from Merck Chemical Company, and purified carbon dioxide (99.98%) and nitrogen (99.995%) as carrier gases were supplied by Wellgas Sdn. Bhd., Malaysia.

#### 2.2. Preparation of the NaOH-AC adsorbent

Commercial coconut shell-based activated carbon was modified by sodium hydroxide (NaOH) impregnation. For this purpose, 10 g of AC was soaked in 100 mL of different strength of aqueous NaOH solutions  $(24-48%)$  and shaken in a water bath shaker for a predetermined dwelling time  $(1-4 h)$  at room temperature and 40 rpm. The sample was then filtered and washed with deionized water. After washing, the sample was dried overnight in an oven at 105 $\degree$ C. The prepared samples were named according to the usage of NaOH% and dwelling time as 24ACSH3, 32ACSH3, 40ACSH3 and 48ACSH3 for 24%, 32%, 40% and 48% NaOH, respectively.

#### 2.3. Carbon dioxide fixed-bed column adsorption procedures

A known amount of adsorbent was placed into the fixed-bed column ( $l = 42$  cm and  $d = 1.1$  cm), and the column was heated to 110  $\degree$ C and held for 1 h under a constant nitrogen flow rate of 90 mL/min to remove excess moisture. At the same time, nitrogen gas and carbon dioxide gas were mixed in a steel mixer and flowed through the analytical system. The inflow of each gas from the cylinders were controlled by a calibrated mass flow controller (AALBORG, model AFC26 NY, USA). After heating, the column was cooled to 35 $\degree$ C. The nitrogen gas purging was stopped, and the adsorption column was vacuumed. Then, the mixed gas was purged upward into the column. The  $CO<sub>2</sub>$  concentration was measured and recorded every 10 s by an online carbon dioxide analyzer model 906 (Quentek instrument, USA). A blank experiment was also performed following the same procedure as described above without the adsorbent in the column and found no  $CO<sub>2</sub>$  adsorption by the column.

The experiment was repeated with different parameters, such as varying reaction temperatures, total gas flow rates, adsorbent loadings and carbon dioxide concentrations, to optimize the reaction conditions. All experiments were conducted under conditions of 0.1 Mpa (atmospheric) pressure and the standard deviation was used as the value of the experimental error.

#### 2.4. Regeneration

The regeneration study was carried out by performing the carbon dioxide adsorption with the sample that gave the optimum result. The first adsorption cycle was performed as mentioned in section 2.3. When the first cycle was completed, the furnace

temperature was increased to 110  $\degree$ C and held for a half hour with nitrogen gas purging. After heating, the temperature was reduced to 35  $\degree$ C and vacuumed. Then, a gas mixture with the same concentration and flow rate as the first cycle was purged upward from the bottom of the column. The concentration of the outlet gas was determined and recorded. The procedures were repeated for a few cycles to determine the reusability of the adsorbent.

#### 2.5. Characterization of the adsorbents

The surface area and porosity properties of the AC and NaOHmodified AC were determined by nitrogen  $(N_2)$  adsorption-desorption at  $-196$  °C (77 K) with a saturation pressure of 106.65 kPa using an automated gas sorption system (Micromeritics, Model ASAP 2020, USA). The BET surface area, Langmuir surface area, cumulative pore volume, micropore volume and average pore width were obtained from the  $N_2$  adsorption isotherms. The external surface area, micropore surface area and micropore volume were evaluated using the t-plot method. The cumulative pore volume was calculated by measuring the amount of  $N_2$  adsorbed at a relative pressure of 0.984. The BJH (Barrett-Joyner-Halenda) method was used to determine the average pore width and pore size distribution.

FT-IR (Fourier transform infrared) spectroscopy analysis was performed using an FT-IR spectrometer (Perkin Elmer, Model 2000 FTIR, USA) to identify the surface functional groups on the AC and NaOH-modified ACs. The surface morphologies of the unmodified and modified AC were examined, and their porosities were verified using a scanning electron microscope (SEM, JEOL JSM- 6460LV, Japan).

#### 3. Results and discussion

#### 3.1. Characterization of the adsorbent

The BET surface areas and average pore sizes of selected adsorbents are presented in Table 1. The values of the BET surface area and micropore area after modification by 32% NaOH decreased drastically compared to the unmodified AC. This observation is attributed to the structural changes of the adsorbents due to the entrapment of NaOH in the micropore area, which reduced the ultimate surface area of the adsorbents during modification with high temperatures [\[17\]](#page--1-0). On the other hand, after the modification, the pore size of AC increased from 2.89 nm to 4.12 nm. The probable mechanism behind the large pore size due to NaOH activation is as follows.

$$
2C + 6NaOH \rightarrow 2Na + 2Na2CO3 + 3H2
$$
 (1)

with elevated temperature,  $Na<sub>2</sub>CO<sub>3</sub>$  degraded into  $CO<sub>2</sub>$  and  $H<sub>2</sub>O$ , which were entrapped and created wider holes and surface become less acidic in presence of  $Na<sup>+</sup>$  by replacing carboxylic and phenols ions [\[18\].](#page--1-0)

[Fig. 1](#page--1-0) shows the SEM images of the unmodified and modified granular activated carbon. The morphological structures appear as





<sup>a</sup> Obtained by BET measurement.

 $b$  Calculated by the BJH (desorption) method using N<sub>2</sub> adsorption isotherms. <sup>c</sup> Obtained by the t-plot method.

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Table 1

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