



# Article Regeneration Performance of Activated Carbon for Desulfurization

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**Abstract:** This study explored the regenerated performance of activated carbon (AC) as SO<sub>2</sub> adsorbent. The optimal conditions of SO<sub>2</sub> removal were determined by experiment, and then the adsorption efficiency of AC was studied by a method of thermal regeneration. The characteristics of regenerated AC were analyzed by Brunauer-Emmett-Teller (BET) and Scanning Electron Microscopy (SEM) methods. The test results showed that the most suitable adsorption conditions were using 4 g of activated carbon, 1.65 L/min gas flue rate, and 5% O<sub>2</sub>. During the ten regenerations, the desulfurization efficiency and sulfur capacity of AC still maintained a high level. The characterization results showed that the increase of material surface area and pore volume were 101 m<sup>2</sup> g<sup>-1</sup>, and 0.13 cm<sup>3</sup> g<sup>-1</sup>, respectively, after the cycles.

Keywords: activated carbons; desulfurization; regeneration

#### 1. Introduction

 $SO_2$  is the major air contaminant released in industrial activities. This gas is mainly generated from the combustion of fossil fuels (coal, oil, and gas), which is for power generation and the smelting of mineral ores, like copper, aluminum, zinc, lead, and iron [1].  $SO_2$  produced by industry can cause acid rain, which contributes to air pollution, soil acidification, water pollution, and destruction of building structures [2]. These environmental problems affect human health, social security, and ecological harmony [3]. Thus, several efficient technologies have been employed for the removal of  $SO_2$  in industrial production by most countries [4].

Porous solid sorbents, such as activated carbon (AC), zeolites, alumina, and zirconia, attract much attention owing to their characteristics of cheap, low energy consumption, and easy regeneration [5,6]. In the area of air pollution control, porous solids have been widely adopted for desulfurization, because of their ultra-low sulfur contents and having high selectivity for sulfur compounds [7]. Specifically, AC is one of the main SO<sub>2</sub> adsorbents, which have abundant oxygen- containing groups, highly porous structure, and good thermal stability [8–10]. The characteristics of abundant oxygen-containing groups and the highly porous structure of AC can enhance the performance for SO<sub>2</sub> physisorption and provide more active sites for SO<sub>2</sub> removal [11,12].

AC adsorbs SO<sub>2</sub> until it reaches its saturation limit. On most occasions, the saturated AC fails to be reused, and is landfilled or incinerated [13]. The regeneration of saturated AC has become a desired feature from academic, industrial, and economic perspectives, as it complies with both the regulations of the discharge of solid pollutants and is good for constructing a sustainable society with a clean and tidy environment [13,14]. The methods of regenerating saturated materials mainly include thermal treatment, oxidation, microwave irradiation, and electrochemical methods [15]. Thermal treatment is the best compromised method in the regeneration of spent adsorbents in the consideration of the cost, the adsorption efficiency, and the number of regeneration cycles. The method is simple and versatile,

and it can obtain better environmental and industrial benefits [14]. A versatile reaction mechanism is shown in Equations (1)–(3) in the process of  $SO_2$  adsorption and desorption by AC:

$$SO_2 + O_2 + H_2O \rightarrow H_2SO_4 \tag{1}$$

$$AC + 2H_2SO_4 \rightarrow CO_2 + 2SO_2 + 2H_2O \tag{2}$$

$$AC + H_2SO_4 \rightarrow CO + SO_2 + H_2O \tag{3}$$

Wang et al. [16] reported an increase in the SO<sub>2</sub> removal efficiency of AC fibers after 1173 K thermal treatment. Saleh et al. [17] showed that the bimetallic nanoparticles which are loaded on the modified AC, can simultaneous remove SO<sub>2</sub>. Jiang et al. [18] revealed that activated carbon obtained from corncob waste showed an effective performance of removal of toluene from flue gas. Li et al. [19] demonstrated that the thermal regeneration of activated coke may cause carbon consumption, which is mostly influenced by the regeneration temperature. However, the performance of AC in the process of multiple recycle regeneration, has been rarely reported.

Thus, this study investigated the influence of various experimental conditions on  $SO_2$  adsorption, and focused both on the investigation of the influence of thermal regeneration on the characteristics of AC, and the adsorption efficiency and mechanism of AC after thermal regeneration [1,2].

#### 2. Materials and Methods

#### 2.1. Materials

AC was supplied by Xinhua Chemical Ltd., China.  $SO_2$  was provided by Shanghai Chunyu Special Gas Co., Ltd. Before the experiment, AC was ground and sieved to 20–40 mesh. Then, AC was dried at 120 °C for 24 h, labeled as AC and stored in a desiccator.

#### 2.2. SO<sub>2</sub> Adsorption Experiment

The experimental device is shown in Figure 1. A single factor experiment was used to investigate the effects of activated carbon dosage, oxygen content, and flue gas flow rate on activated carbon adsorption, as shown in Table 1. A certain amount of adsorbents were placed in a 15 × 150 mm U- shaped tube and heated in a water bath at 60 °C, under a mixed gas flow (the consistent mixed gas contained 2000 ppm SO<sub>2</sub>, a certain amount of O<sub>2</sub>, and was balanced by N<sub>2</sub>. The amount of SO<sub>2</sub> in the exhaust gas was analyzed by a portable flue gas analyzer (KANE9506, England KANE). The desulfurization efficiency of the adsorbent AC calculated, which determined the suitable adsorption conditions. The desulfurization efficiency can be evaluated by:

$$\eta = \frac{C_i - C_o \times 100\%}{C_i} \tag{4}$$

where  $\eta$  is the desulfurization efficiency, Ci represents the SO<sub>2</sub> inlet concentration, and Co the SO<sub>2</sub> outlet concentration (ppm).



Figure 1. Schematic of the experimental apparatus.

AC Dosage/g	Flue Gas Rate/L∙min <sup>-1</sup>	Oxygen Content/%
2	1.65	5
4	1.65	5
6	1.65	5
8	1.65	5
10	1.65	5
4	1	5
4	2	5
4	3	5
4	4	5
4	1.65	0
4	1.65	2.5
4	1.65	5
4	1.65	7.5

Table 1. Experimental conditions of SO<sub>2</sub> adsorption.

## 2.3. Recycle of AC

Saturated AC was placed in a quartz boat and heated in a tube furnace at 420 °C for 1.5 h, under a gas flow rate of 1.65 L/min. The AC was weighed after cooling to room temperature. After that, the regenerated adsorbent was treated to remove  $SO_2$  cyclically under the suitable experimental conditions, while the cycles were carried out ten times. The regenerated samples were marked as Reg-ACn, where n represents the number of cycles. After each regeneration, the weights of the adsorbent were recorded, and the regeneration loss and sulfur capacity calculated. The samples were characterized by several methods, and the desulfurization efficiency and adsorption capacity obtained. The adsorption capacity can be calculated as follows:

$$q_m = F/m \left( C_i t - \int_0^t C_o dt \right)$$
(5)

where  $q_m$  is the adsorption capacity of SO<sub>2</sub> (mg/g); F is the flue gas rate (L/min); Ci represents the SO<sub>2</sub> inlet concentration, and Co the SO<sub>2</sub> outlet concentration (ppm); m is the weight of AC (g); t is the time of removal of SO<sub>2</sub> (min).

#### 2.4. Characterization of AC

The SEM images of AC were obtained using a scanning electron microscope (SEM, JSM–6460, Japan JEOL). The textural characteristics of the AC samples were analyzed by adsorption–desorption of nitrogen at 77 K on a Micromeritics ASAP 2020 auto-adsorption system (Norcross, GA, USA).

#### 3. Results and Discussion

#### 3.1. Characterization of AC

#### 3.1.1. SEM Analysis

The SEM images of the six AC samples after five times regeneration are shown in Figure 2a–f. From the aspects of pore structure, the surface of the samples appeared to have a structure with more micropores and mesoporous (Figure 2b) after thermal regeneration. Additionally, some micropore structures were observed within the macropores as in Figure 2e,f. After the 8th cycle, the pores were irregularly arranged and partly collapsed. Comparing the surface morphology of the AC before and after desulfurization, some white particles with irregular shape were deposited on the surface of the samples after regeneration, which could have been sulfate. Some of the micropores became clogged with these particles. The results indicated that new micropores are generated after regeneration, which is conducive to the adsorption of  $SO_2$  by AC, while the deposition of sulfate lead to a decline in desulfurization activity with continued cycles of regeneration.



**Figure 2.** SEM image of the prepared activated carbon (AC) treated with regeneration after 0 (**a**), 2 (**b**), 4 (**c**), 6 (**d**), 8 (**e**), 10 (**f**) number of cycles.

#### 3.1.2. BET Analysis

As shown in Table 2, the micropore area accounts for approximately 3/4 of the total area, which suggests that the samples mainly contained micropores. The micropore area, surface area, and pore volumes were slightly increased after thermal regeneration, owing to the continual etching of the

formed micropores [20]. This case favored the improvement of the storage capacity for  $H_2SO_4$  and the desulfurization efficiency of AC.

Cycles	Micropore Area(m <sup>2</sup> /g)	BET Surface(m <sup>2</sup> /g)	Total Pore Volume(cm <sup>3</sup> /g)	Average Pore Size(nm)
0	356.449	463.601	0.246	2.594
2	387.726	498.530	0.273	3.074
4	395.423	518.272	0.291	3.299
6	435.656	563.622	0.321	3.229
8	463.761	589.350	0.319	5.027
10	443.978	664.601	0.373	2.793

Table 2. Surface area and pore volume analysis.

 $N_2$  adsorption–desorption isotherms and pore size distribution of all samples are depicted in Figure 3. In Figure 3a, the presence of micropores was proved owing to the dramatic increase of the adsorbed volume, when the adsorbents are at low relative pressure (P/P0 < 0.2) [21,22]. According to IUPAC classification [23], the adsorption–desorption isotherms of all samples comply with typical I-type isotherms of microporous solids. A narrow and long hysteresis ring appeared in the curve at relative high pressure, and which ended at P/P0 = 1. This indicates that the samples contained both micropores and mesopores. In Figure 3b, the broadening of pore size distribution of AC after thermal regeneration was shown from the distribution of the pore size obtained by the Barrett-Joyner-Halenda (BJH) methods [20,24], which provides evidence for a slight increase of the surface area and pore volumes. Many studies suggested that the adsorption capacity is mainly affected by the micropores and mesopores and mesopores in the use of the adsorption capacity is mainly affected by the micropores and mesopores exist simultaneously in the solid adsorbent [25].



**Figure 3.** The N<sub>2</sub> sorption isotherms (**a**) and pore size distributions (**b**) of the samples (where AC. stands for fresh AC, Reg-ACn stands for the nth regenerated AC).

#### 3.2. SO<sub>2</sub> Adsorption

# 3.2.1. Effect of AC Dosage on $SO_2$ Adsorption

As seen in Figure 4, at amounts of 2 g, 4 g, 6 g, 8 g, and 10 g, the average  $SO_2$  adsorption rates of AC in ten minutes were 42.3%, 55.8%, 62.3%, 68.5%, and 79.7%, respectively. The desulfurization efficiency is improved by the increase of the AC amount. This indicates that the  $SO_2$  saturation adsorption capacity of the samples is highly influenced by the amount of AC. On increase of dosage, more active

surface areas and sites are provided from the AC, which develops the removal capacity of  $SO_2$  [26]. The best option combines both the cost and desulfurization efficiency, so we fixed the AC dosage for the rest of the experiment at an amount of 4 g.



**Figure 4.** Effect of change of carbon dosage on SO<sub>2</sub> adsorption behavior (operated at 60 °C, for 2, 4, 6, 8, 10 g samples, 2000 ppm SO<sub>2</sub>, 5% O<sub>2</sub>, N<sub>2</sub> to balance and 1.65 L/min total flow rate).

#### 3.2.2. Effect of Flue Gas Flow Rate on SO<sub>2</sub> Adsorption

The influence of gas flow rate on adsorption performance was studied by analysis of the desulfurization efficiency at flow rates of 1, 2, 3, and 4 L/min. As seen in Figure 5, the average SO<sub>2</sub> adsorption rates of AC at the gas flow rate of 1 L/min, 2 L/min, 3 L/min and 4 L/min were 86.2%, 75.9%, 62.3%, and 57.4% in ten minutes, respectively. The slower the gas flow rate, the higher is the adsorption efficiency. It is evident that the faster the gas flow rate, the lower the retention time of the SO<sub>2</sub> in the AC of the U-shaped tube, thus the adsorption efficiency decreases. In addition, as the gas flow rate decreases, the adsorption efficiency gradually becomes improved. The reason for this phenomenon is that the lower the flue gas rate, the bigger is the resistance [27]. So for the other experiments, the flue gas flow rate of 1.65 L/min as a fixed condition.



**Figure 5.** Effect of flue gas flow rate on SO<sub>2</sub> adsorption (operated at 60 °C, 4 g sample, 2000 ppm SO<sub>2</sub>, 5% O<sub>2</sub>, N<sub>2</sub> to balance and 1, 2, 3, 4 L/min total flow rate).

#### 3.2.3. Effect of Oxygen Content on SO<sub>2</sub> Adsorption

To investigate the influence of  $O_2$  contents on the SO<sub>2</sub> adsorption efficiency, experiments were carried out under conditions of different  $O_2$  content (0, 2.5, 5, and 7.5%  $O_2$ ). Below 5%, it was observed from Figure 6 that with the increase in  $O_2$  content, the desulfurization efficiency increases. It indicated that the present of  $O_2$  could improve the desulfurization efficiency. This can be explained as follows: if oxygen and water vapor exist in the process of SO<sub>2</sub> adsorption, SO<sub>2</sub> will be oxidized to sulfuric acid and introduced into the pores of the AC, which improves the desulfurization efficiency of AC [28]. When the pore widths in the range of microporous to mesoporous increase, the capacity for storing gas and liquid may be improved [20]. The desulfurization efficiency above 5% was less. It could be shown that a large number of oxides were produced due to the combination of too much  $O_2$  with the active components on the AC surface, thereby the AC fails to combine with more active sites, thereby its desulfurization efficiency decreases [29]. Therefore, we selected a fixed condition of oxygen content of 5% for the rest of the experiments.



**Figure 6.** Effect of change in oxygen content on SO<sub>2</sub> adsorption behavior (operated at 60 °C, 4 g sample, 2000 ppm SO<sub>2</sub>, N<sub>2</sub> to balance, and 1 L/min total flow rate).

#### 3.3. AC Regeneration

The SO<sub>2</sub> breakthrough curves after ten adsorption desorption cycles are illustrated in Figure 7. Under suitable adsorption conditions (4 g AC, 1.65 L/min flow gas, and 5% O<sub>2</sub>), the average desulfurization rate of fresh AC over the first ten minutes is about 96%, and the highest desulfurization rate of all the samples achieves more than 90%. The desulfurization efficiency decreased gradually after multiple regenerations. First, one of the reasons maybe that the micropores became disrupted after thermal treatment. This problem can be minimized in mesoporous carbon materials, such as porous carbon nanofibers, owing to their thermal and mechanical stability [30]. Second, the surface of AC produced some sulfates or oxygen groups in the regeneration [31,32], a part of the micropores of AC was blocked after this situation. In addition, a certain amount of carbon loss may occur during the process of thermal treatment. Despite this, the highest desulfurization efficiency of 95.4% was still achieved at the tenth cycle.



**Figure 7.** SO<sub>2</sub> desulfurization efficiency of the regenerated adsorbents, where 0–10 stands for number of cycles.

As shown in Figure 8, the sulfur capacity of fresh AC is 38.88 mg/g. After the first heating regeneration at 400 °C, the decrease in sulfur capacity is most obvious. Though the SO<sub>2</sub> capacity of AC decreases continuously with multiple cycles, the regenerated AC after 10 cycles showed a sulfur capacity of 23.44 mg/g for SO<sub>2</sub> adsorption, which only decreased by 15.44 mg/g compared to that of fresh AC, while after the seventh time, the rate of sulfur capacity decrease became slower. Zhang et al. [15] showed that the adsorption capacity of CuFe<sub>2</sub>O<sub>4</sub>/AC magnetic adsorbent is high, but the adsorption capacity of CuFe<sub>2</sub>O<sub>4</sub>/AC decreases significantly after regeneration. According to Xu et al. [33] biochar made from cow manure, sewage sludge, and rice husks showed an SO<sub>2</sub> adsorption capacity of up to 64 mg g<sup>-1</sup>, but its specific surface area was too low (the maximum was 42 m<sup>2</sup> g<sup>-1</sup>). In comparison, the adsorption capacity of commercial AC in the regeneration process did not significantly decrease. The reason is that in the process of sulfuric acid etching, the pore widths in the range of microporous to mesoporous increased, which improved the capacity for storing sulfuric acid, and new micropores were produced in the process of sulfuric acid etching. Those conditions are good for the recovery of the porous volume and adsorption capacity of AC after the cycles of regeneration.



Figure 8. Sulfur capacity of the regenerated adsorbents, where 0–10 stands for number of cycles.

## 4. Conclusions

The effect of  $SO_2$  adsorption efficiency was experimentally studied with different carbon dosages, flue gas flow rates, and oxygen contents. Then, the characterization and desulfurization efficiency of AC was observed under the current optimum adsorption conditions for a number of 10 heated regeneration cycles. During the thermal regenerations, micropores became disrupted, and some of

them became blocked by sulfate particles on the AC surface. However, owing to sulfuric acid etching, the pore widths in the range of microporous to mesoporous increased and some new micropores were produced. Therefore, the specific surface area and pore volume of AC were slightly increased. The highest desulfurization efficiency of fresh AC was 98.7%. The highest desulfurization efficiency was 95.4% after ten cycles. The results show that AC has a high SO<sub>2</sub> adsorption efficiency and the performance of desulfurization is only slightly decreased after ten thermal regeneration cycles, so it can be concluded that AC is a suitable material for multiple adsorption–desulfurization cycles.

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