



# Article Elemental Mercury Adsorption by Cupric Chloride-Modified Mesoporous Carbon Aerogel

Dongjing Liu <sup>1</sup>, Cheng Lu <sup>2</sup> and Jiang Wu <sup>2,\*</sup>

- <sup>1</sup> School of Energy and Power Engineering, Jiangsu University, Zhenjiang 212013, China; liudongjing19@163.com
- <sup>2</sup> College of Energy and Mechanical Engineering, Shanghai University of Electric Power, Shanghai 200090, China; luchengsuep@163.com
- \* Correspondence: wujiang207@163.com or wujiang@shiep.edu.cn; Tel./Fax: +86-02135303902

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**Abstract:** Mesoporous carbon aerogels (MCA) synthesized via aqueous polymerization of resorcinol and formaldehyde were modified by copper chloride and applied for adsorption removal of elemental mercury (Hg<sup>0</sup>) at a low temperature. The sorbents were characterized by field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), nitrogen adsorption/desorption isotherms, and X-ray photoelectron spectroscopy (XPS) techniques. The results show that CuCl<sub>2</sub>-modified MCA exhibited good ability for Hg<sup>0</sup> adsorption in the 40–160 °C temperature range, with Hg<sup>0</sup> removal efficiency all above 95.0%. The Hg<sup>0</sup> removal efficiency first increased and then decreased with the elevation of reaction temperature. It performed optimally at 80 °C with the highest Hg<sup>0</sup> removal efficiency of 98.7%. XPS results indicate that covalent chlorines (C-Cl groups) play an important role in elemental mercury adsorption process. Hg<sup>0</sup> is firstly captured in the form of oxidized mercury (Hg<sup>2+</sup>) and then reacts with C-Cl groups to form HgCl<sub>2</sub>.

Keywords: elemental mercury; carbon aerogel; covalent chlorine; copper chloride

## 1. Introduction

Mercury is one of the most toxic heavy metal elements in the environment due to its high volatility, long persistence, and strong bioaccumulation property [1]. Coal-fired power plants are the dominant anthropogenic mercury emission sources [2]. Elemental mercury, oxidized mercury, and particle-bound mercury are three types of mercury species in flue gas [3]. On account of its low aqueous insolubility and high vaporability, elemental mercury (Hg<sup>0</sup>) capture from flue gas is considered the hardest and most urgent work in the mercury emission control of coal-fired power plants [4].

Adsorption removal of elemental mercury by using solid sorbents is a viable method with respect to mercury emission control of coal-fired power plants [5]. Enormous carbon-based or noncarbon-based materials, e.g., biomass-derived chars [6], activated carbon [7], fly ash [8], as well as natural minerals [9], had been extensively studied for elemental mercury removal from flue gas. However, most of them exhibit limited mercury adsorption capacity and poorer Hg<sup>0</sup> removal efficiency at low temperatures under flue gas free of HCl [10]. Carbon aerogels consisting of interconnected and uniform particles with well-defined pore architectures are promising sorbents for elemental mercury capture [11]. The large surface area and high porosity of carbon aerogels could offer abundant active sites for Hg<sup>0</sup> adsorption and benefits a mass transfer process (e.g., Hg<sup>0</sup> vapor diffusion) [12]. CuCl<sub>2</sub>-modification is found to be effective for mercury adsorption, and the chlorine ions contained in CuCl<sub>2</sub> are favorable for Hg<sup>0</sup> oxidation in chlorine-free flue gas [13]. In addition, CuCl<sub>2</sub> possesses the advantage of lower capital cost compared with the commonly used metal oxide additives. Yang et al. [14] developed a CuCl<sub>2</sub>-modified magnetosphere for elemental mercury removal. It performed optimally at 150 °C with the highest Hg<sup>0</sup> removal efficiency of 90.6%. Zhou et al. [15] synthesized a CuCl<sub>2</sub>-impregnated CeO<sub>2</sub>-ZrO<sub>2</sub> sorbent for  $Hg^0$  removal in chlorine-free coal combustion flue gas. They revealed that the interaction between Cl and chemical adsorbed oxygen contributed to its superior and stable mercury capture performance with a  $Hg^0$  removal efficiency of 89.6–97.1%. Therefore, CuCl<sub>2</sub> is employed as the additive for carbon aerogels for the sake of combining the prominent oxidation ability of CuCl<sub>2</sub> and the good mass transfer ability of carbon aerogels toward elemental mercury adsorption.

In this work, mesoporous carbon aerogels (MCA) with large pore volume (>1 cm<sup>3</sup>/g) and large specific surface area (>500 m<sup>2</sup>/g) were synthesized via the sol-gel method. The as-prepared MCA was subsequently modified with CuCl<sub>2</sub> by using the pore volume impregnation approach for Hg<sup>0</sup> adsorption at an upflow bench-scale fixed-bed reactor in the temperature range of 40–160 °C. The samples were characterized by using field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), nitrogen adsorption/desorption, and X-ray photoelectron energy spectroscopy (XPS) techniques. The effects of CuCl<sub>2</sub> concentration and reaction temperature on Hg<sup>0</sup> adsorption performances of CuCl<sub>2</sub>-modified MCA were investigated. Furthermore, the reaction mechanism of Hg<sup>0</sup> capture over CuCl<sub>2</sub>-modified MCA is also addressed according to the XPS analysis results. Cu06MCA, Cu18MCA, and Cu30MCA denote the MCA modified by CuCl<sub>2</sub> with molar concentrations of 0.06, 0.18, and 0.30 mol/L, respectively.

## 2. Results and Discussions

#### 2.1. Characterization Analysis

The FESEM images of pristine and CuCl<sub>2</sub>-modified MCA are shown in Figure 1. The as-prepared MCA was formed by interconnected irregular spherical particles (Figure 1a), and many pores were produced among the particles which aggregated together. After modification with cupric chloride, the surface of the MCA became a little bit compact, and the MCA particles cohered together (Figure 1b), probably leading to the reduction of the Brunauer-Emmett-Teller (BET) surface area (Table 1).



**Figure 1.** Field emission scanning electron microscopy (FESEM) images: (**a**) Mesoporous carbon aerogels (MCA) and (**b**) Cu18MCA.

The XRD patterns of pristine and  $CuCl_2$ -modified MCA are presented in Figure 2. The bigger broad diffraction peaks at ~23.9° were assigned to the reflection of the (002) lattice plane of activated carbon and graphite. The smaller broad diffraction bands at ~43.6° were attributed to the reflection of the (100) lattice plane of graphite [16,17]. The signals of CuCl<sub>2</sub> were not detected in CuCl<sub>2</sub>-modified MCA, probably owing to the smaller content of CuCl<sub>2</sub> or the thorough dispersion of amorphous CuCl<sub>2</sub> on the MCA surface.



Figure 2. X-ray diffraction (XRD) patterns of pristine and CuCl<sub>2</sub>-modified MCA.

The nitrogen adsorption–desorption isotherms and pore size distributions of pristine as well as  $CuCl_2$ -modified MCA are displayed in Figure 3. The MCA exhibited type IV isotherm (Figure 3a). The position of the hysteresis loop at the higher relative pressure of above 0.8 implies the presence of large mesopores on the MCA surface [18], which is consistent with the broad pore size distribution centered between 10 and 30 nm. The BET surface area, total pore volume, and pore diameter of the MCA were 523 m<sup>2</sup>/g, 1.21 cm<sup>3</sup>/g, and 9.2 nm, respectively (Table 1). After modifying with CuCl<sub>2</sub>, the mesoporous structures of the MCA were still preserved. However, the pore size distribution became narrow, with pore diameter centered at ~16 nm (Figure 3b).



**Figure 3.** (a) Nitrogen adsorption–desorption isotherms and (b) pore size distributions of pristine and CuCl<sub>2</sub>-modified MCA.

Table 1.	Textural	property	y of	pristine and	CuCl <sub>2</sub> -r	nodified	MCA.
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Samples	Surface Area (m²/g)	Total Pore Volume (cm <sup>3</sup> /g)	Micropore Volume (cm <sup>3</sup> /g)	Mesopore Volume (cm <sup>3</sup> /g)	Pore Diameter (nm)
MCA	523	1.21	0.15	1.06	9.2
Cu06MCA	641	1.38	0.06	1.32	8.6
Cu18MCA	474	1.12	0.12	1.00	9.5
Cu30MCA	413	1.09	0.11	0.98	10.6

The XPS spectra of fresh and spent Cu18MCA are displayed in Figure 4 to determine the surface species on sorbent surfaces. The C 1s spectra can be deconvoluted into five components in which chemical shifts are assigned to carboxyl groups (~288.6 eV), C=O bonds (~287.0 and ~287.2 eV), C-OH groups (~285.4, ~285.6, and 286.2 eV), and graphitic carbon (sp<sup>2</sup> configuration: ~284.2 and ~284.8 eV) [18]. In addition, the broad band at ~290.0 eV is related to shake-up satellite peaks assigned to  $\pi$ - $\pi$ \* transitions in aromatic rings [19]. The Cu 2p peak of fresh Cu18MCA at ~935.8 eV is assigned

to the reflection of  $Cu^{2+}$  ions in  $CuCl_2$  [20]. It shifts slightly toward higher values of 936.2 eV after  $Hg^0$  adsorption, possibly attributed to the formation of an oxychloride phase  $Cu_2OCl_2$  [21], which is an important intermediate for mercury oxidation. As for spent Cu18MCA, the signal at ~105.4 eV is due to the reflection of Hg 4f 5/2 orbit of oxidized mercury, implying that Hg<sup>0</sup> transformed into  $Hg^{2+}$  during the mercury adsorption process [22]. The peaks at ~200.2 and ~202.4 eV belong to covalent chlorine (C-Cl groups), while the bands at ~198.2 and ~199.8 eV are ascribed to ionic chlorine (Cl<sup>-</sup> ions) [23]. The content of the covalent chlorine (C-Cl groups) decreased from 81.3% to 47.8% for fresh and spent Cu18MCA, respectively, indicating that some C-Cl groups transformed into Cl<sup>-</sup> ions during the mercury adsorption process [24]. Since the mercury on the surface of spent Cu18MCA exists in an oxidation state (Hg<sup>2+</sup>), the adsorbed mercury on the sorbent surface may react with C-Cl groups to form HgCl<sub>2</sub>, and therefore the reaction mechanism of elemental mercury reacting with covalent chlorine (C-Cl groups) can be summarized as follows [25]: (i) Cl<sup>-</sup> ions combined with carbon atoms to form C-Cl groups; (ii) gaseous Hg<sup>0</sup> was oxidized by Cu<sub>2</sub>OCl<sub>2</sub> and captured on the sorbent surface in the form of oxidized mercury (Hg<sup>2+</sup>); and (iii) finally, the adsorbed mercury reacted with the C-Cl groups to generate HgCl<sub>2</sub>.



**Figure 4.** X-ray photoelectron spectroscopy (XPS) spectra of (**a**, **c**, **e**) fresh Cu18MCA and (**b**, **d**, **f**, **g**) spent Cu18MCA.

## 2.2. Effect of CuCl<sub>2</sub> Concentration

The impact of CuCl<sub>2</sub> concentration on Hg<sup>0</sup> adsorption performance at 120 °C is presented in Figure 5. Pristine MCA showed weaker Hg<sup>0</sup> adsorption ability. Though the Hg<sup>0</sup> removal efficiency could reach as high as ~88.0% within 2 min, it dropped rapidly with the time elapsed and maintained at only ~27.3% after mercury adsorption over 80 min. The probable reason is that Hg<sup>0</sup> adsorption on MCA is a physisorption process, and the number of the active sites of MCA will be consumed during mercury adsorption process, leading to the rapid decrease in Hg<sup>0</sup> removal efficiency. However, the Hg<sup>0</sup> capture ability of MCA can be effectively improved by loading with CuCl<sub>2</sub>. The CuCl<sub>2</sub>-modified MCA performed well toward Hg<sup>0</sup> adsorption, with Hg<sup>0</sup> removal efficiency all above 96.0% due to the

addition of new chemisorption sites (Cu<sub>2</sub>OCl<sub>2</sub> and C-Cl group) [26]. The Hg<sup>0</sup> removal efficiency of MCA significantly rose to ~97.3% and ~97.6% when CuCl<sub>2</sub> concentrations were 0.06 mol/L and 0.18 mol/L, respectively. However, it dropped slightly to ~96.3% when CuCl<sub>2</sub> concentration further increased from 0.18 to 0.30 mol/L. According to the nitrogen adsorption–desorption data, when CuCl<sub>2</sub> concentration was 0.30 mol/L, BET surface area and pore volume greatly decreased thereby weakening the mercury adsorption process because excessive CuCl<sub>2</sub> will deteriorate the pore structures of the MCA. Thus, the optimal CuCl<sub>2</sub> concentration is 0.18 mol/L.



**Figure 5.** Effect of CuCl<sub>2</sub> concentration on Hg<sup>0</sup> adsorption performance of MCA at 120 °C.

#### 2.3. Effect of Reaction Temperature

The impact of reaction temperature on  $Hg^0$  adsorption performance of Cu18MCA is presented in Figure 6. Cu18MCA performed well toward  $Hg^0$  adsorption in the 40–160 °C temperature range, with  $Hg^0$  removal efficiency all above 95.0%, indicating that  $Hg^0$  adsorption on CuCl<sub>2</sub>-modifed MCA was mainly dominated by the chemisorption process. The  $Hg^0$  sorption rate at the initial reaction stage and the equilibrium of  $Hg^0$  removal efficiency first increased and then decreased with the reaction temperature rising from 40 to 160 °C. Cu18MCA performed the best at 80 °C with the fastest  $Hg^0$  sorption rate and the highest  $Hg^0$  removal efficiency of ~98.7%. Though higher temperatures can enhance chemisorption processes [27], the product mercury chloride will decompose at elevated temperatures, resulting in the decline of  $Hg^0$  removal efficiency. Thus, the optimal reaction temperature for Cu18MCA is 80 °C. In addition, Cu18MCA performance was fairly stable over 600 min on stream, with  $Hg^0$  removal efficiency staying above 98.0% all times (Figure 7).



Figure 6. Effect of reaction temperature on Hg<sup>0</sup> adsorption performance of Cu18MCA.



**Figure 7.** Hg<sup>0</sup> adsorption performance of Cu18MCA at 120 °C on stream over 600 min.

## 3. Experimental

#### 3.1. Catalyst Synthesis and Characterization

Mesoporous carbon aerogels (MCA) were synthesized via the aqueous polymerization of resorcinol and formaldehyde [28]. CuCl<sub>2</sub>-modified MCA was fabricated via the incipient wetness impregnation method. First, certain amounts of CuCl<sub>2</sub>·2H<sub>2</sub>O were dissolved in 3.0 g of deionized water to obtain solutions with CuCl<sub>2</sub> molar concentrations of 0.06, 0.18, and 0.30 mol/L, respectively. Then, about 1.0 g of as-prepared MCA was added into the aforementioned solution and subsequently dried at 70 °C overnight. The resultant sorbents were denoted as Cu06MCA, Cu18MCA, and Cu30MCA, respectively. The FESEM images were detected on a Phillips XL-30 FEG/NEW instrument (Eindhoven, The Netherlands). The XRD patterns were measured on Bruker D8 Advance equipment (Karlsruhe, Germany). The nitrogen isotherms were examined at 77 K on a Beishide 3H-2000PS4 apparatus (Norcross, GA, USA). The pore size distributions were estimated with the Barrett-Joyner-Halenda (BJH) method using the desorption branch of N<sub>2</sub> isotherm, which is expressed by the Kelvin equation as follows:

$$\ln(p^*/p_0) = -\left(\frac{2\gamma\nu\cos\theta}{RTr_m}\right) \tag{1}$$

where  $p^*$  is the condensing pressure,  $p_0$  is the saturated vapor pressure,  $\gamma$  is the liquid surface tension,  $\nu$  is the adsorbate molar volume,  $\theta$  is the angle between the liquid and solid surface, and  $r_m$  is the mean radius of curvature of the liquid meniscus. XPS analyses were carried out on a RBD upgraded PHI-5000C ESCA system (Perkin Elmer, Waltham, MA, USA) with Mg K $\alpha$  radiation ( $h\nu$  = 1253.6 eV) or Al K $\alpha$  radiation ( $h\nu$  = 1486.6 eV).

#### 3.2. Mercury Adsorption

The mercury adsorption testing system has been described in detail elsewhere [29,30]. About 50 mg of sorbents were loaded into a fixed-bed reactor and heated up to the desired temperatures. Then, the nitrogen stream containing  $Hg^0$  vapor produced by a mercury generator at 50 °C was consecutively charged into the reactor.  $Hg^0$  concentration was measured by an on-line mercury analyzer (Lumex, RA-915-M, St. Petersburg, Russia). The mercury conversion can be attained by

$$\eta = (1 - C_{out} / C_{in}) \times 100\%$$
<sup>(2)</sup>

where  $\eta$  denotes the mercury removal efficiency (%), and  $C_{in}$  and  $C_{out}$  denote the inlet and outlet mercury concentration ( $\mu g/m^3$ ), respectively.

## 4. Conclusions

CuCl<sub>2</sub>-modified MCA exhibited excellent  $Hg^0$  adsorption performance in the temperature range of 40–160 °C. The  $Hg^0$  removal performance of Cu18MCA first ascended and then descended with rising reaction temperatures. It possessed the highest  $Hg^0$  removal efficiency of ~98.7% and the fastest  $Hg^0$  sorption rate at 80 °C. Chemisorption dominated the mercury adsorption process over CuCl<sub>2</sub>-modified MCA. Covalent chlorine (C-Cl groups) played a crucial role in elemental mercury adsorption process.  $Hg^0$  was first oxidized by Cu<sub>2</sub>OCl<sub>2</sub> and captured in the form of oxidized mercury (Hg<sup>2+</sup>) and then reacted with C-Cl groups to form HgCl<sub>2</sub>.

Author Contributions: D.L. designed the experiments, analyzed the data, and wrote the paper. C.L. performed the experiments and characterized the samples. J.W. conceived the project.

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Conflicts of Interest: The authors declare no conflict of interest.

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Sample Availability: Samples of the compounds are not available from the authors.



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