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Adsorption enhancement of elemental mercury by various surface modified coconut husk as eco-friendly low-cost adsorbents



Khairiraihanna Johari^a, Norasikin Saman^b, Shioh Tien Song^b, Cheu Siew Chin^b, Helen Kong^b, Hanapi Mat^{b, c, *}

^a Chemical Engineering Department, Universiti Teknologi PETRONAS, 32610, Bandar Seri Iskandar, Perak, Malaysia

^b Advanced Materials and Process Engineering Laboratory, Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor, Malaysia

^c Advanced Material and Separation Technologies (AMSET) Research Group, Health and Wellness Research Alliance, Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor, Malaysia

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ABSTRACT

Coconut husk (CH), consisting of coconut pith (CP) and coconut fiber (CF) is abundant and cheap, and has the potential to be used as adsorbent for elemental mercury (Hg^0) removal. CP and CF surfaces were modified by mercerization and bleaching methods and characterized using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and analysis of moisture and ash. The elemental mercury adsorption measurements were carried out at the following conditions: initial Hg^0 concentration, $200 \pm 20 \mu\text{g}/\text{m}^3$; bed temperature, $50 \pm 1 \text{ }^\circ\text{C}$; N_2 flow rate, 0.05 L/min; mass of adsorbent, 50 mg; and adsorbent particle size of between 75 and 100 μm . The surface morphology and surface functional groups of adsorbents significantly changed after treatments and resulted in different Hg^0 adsorption performances. The highest Hg^0 adsorption capacity was observed for CP-NaOH (956.282 ng/g), followed by CP-Pristine (730.250 ng/g), CF-NaOCl (639.948 ng/g), CF- H_2O_2 (634.347 ng/g), CF-NaOH (611.678 ng/g), CF- H_2O_2 (531.277 ng/g), CP-NaOCl (501.126 ng/g), and CF (431.773 ng/g). The experimental breakthrough data for all the adsorbents produced a good fit to the pseudo-second order kinetic model.

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1. Introduction

Mercury is one of the most toxic heavy metals, which can contaminate the environment and accumulate in animals and plants (Wang et al., 2014). Mercury may exist in three different forms namely metallic mercury (e.g. Hg^0), inorganic mercury compounds (e.g. HgCl_2) and organic mercury compounds (e.g. MeHg^+). These mercury species are mostly released into the atmosphere by natural processes such as volcanic eruptions and geothermic activities; as well as from anthropogenic sources such as coal power plants, metal mining and refining, cement plants, municipal incinerators, and wellhead natural gas processing (Suresh Kumar Reddy et al., 2014; Wang et al., 2014).

The flue gas from coal-fired power plants comprises mercury and acid gases such as NO_x and SO_2 at elevated temperatures. It is the largest source of mercury emission. The removal of Hg^0 is difficult because of its insolubility in water, high volatility and chemical inertness (Padak and Wilcox, 2009). In air pollution control devices (APCD) such as wet scrubbers, elemental mercury removal is achieved by the direct upstream injection of adsorbent into the flue gas stream. At the tail-end (downstream) of the particulate control devices after a fabric filter or electrostatic precipitator, the gas stream still contains low concentrations of NO_x ($<200 \text{ mg}/\text{Nm}^3$), SO_2 ($<100 \text{ mg}/\text{Nm}^3$), dust and residual elemental mercury. The residual elemental mercury can be further removed from the gas stream with a fixed-bed adsorber system (Wilcox et al., 2012; Xu et al., 2014). The mercury also exists in natural gas as an impurity at concentrations sufficiently high to cause both safety and health concerns (Rashid et al., 2013). It was reported that the elemental mercury can amalgamate with aluminum and damage heat exchangers causing mechanical

* Corresponding author. Advanced Materials and Process Engineering Laboratory, Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor, Malaysia.

E-mail address: hmat@cheme.utm.my (H. Mat).

failure and gas leakage (Rashid et al., 2013; Wilhelm and Bloom, 2000).

The removal of elemental mercury from gas stream has been the subject of intensive research over the years. Adsorption based processes are effective for removing elemental mercury from flue and natural gases. Their successful application depends, among others on good adsorption characteristics and cheaper adsorbents. Activated carbon is undoubtedly the most popular commercial adsorbent for elemental mercury removal applications (Hsi and Chen, 2012), but it requires the addition of complexing agents such as sulfur to improve its elemental mercury removal performance (Granite et al., 2000; He et al., 2012). Other successful adsorbents which are commercially used in removing mercury from natural gas are silver modified zeolite molecular sieves, alumina, oxides and polymer resins (Wilhelm and Bloom, 2000). The limitations of the existing adsorbents such as high capital and operational cost have led to research into other possible elemental mercury adsorbents. This includes the use of fly ash (Serre and Silcox, 2000) calcium-based adsorbents (Ghorishi and Sedman, 1998), oxides (Zhou et al., 2013), and functionalized silica (Johari et al., 2014a,b; Saman et al., 2015).

In the last two decades, the development of low-cost adsorbents based on lignocellulosic agricultural wastes has gained momentum. It has been demonstrated that residues from oil palm (*Elaeis Guineensis*), rice (*Oryza Sativa L.*) and coconut (*Cocos Nucifera L.*) production have high adsorption capacity towards heavy metals from wastewaters (Bhatnagar et al., 2010; Johari et al., 2013, 2014a,b; Sharma et al., 2013; Song et al., 2013). These agricultural wastes would be attractive as precursors for development of adsorbents due to their abundance and low cost, and simple preparation process (Bhatnagar et al., 2010; Johari et al., 2013, 2014a,b; Sharma et al., 2013; Song et al., 2013). As reported in many previous studies, the unprocessed precursors exhibit a lower adsorption capacity, therefore several treatments and functionalization methods have been investigated in order to overcome this drawback, as well as to enhance their selectivity and regenerability (Johari et al., 2014a,b; Song et al., 2013). Alkaline hydrolysis using sodium hydroxide or oxidative bleaching delignification using sodium hypochlorite, sodium chlorite or hydrogen peroxide have been reported effective at increasing exposed cellulose surface area, thus increasing the number of reaction sites (Kumar et al., 2009; Sreekala et al., 1997).

It was previously reported that the use of coconut wastes (e.g. desiccated, pith, fiber and shell) as potential adsorbents for the removal of diverse heavy metal ions, dyes, inorganic anions, radionuclides, and miscellaneous pollutants (Bhatnagar et al., 2010; Isah et al., 2015; Johari et al., 2014a,b; Mohammed et al., 2015) from water showed very encouraging results. However, the use of coconut waste available in large quantity at low cost, especially coconut husk as adsorbents for elemental mercury (Hg^0) removal is still limited. Furthermore, the surface modification of the coconut husk (pith and fiber) for optimization of elemental mercury adsorption has not been thoroughly reported. This study aims to evaluate the potential use of pristine and surface-treated coconut husk (pith and fiber) adsorbents for removing elemental mercury from gases with low acidity and temperature typically encountered downstream of flue gas particulate control devices or in natural gas processing applications carried out in a fixed-bed adsorber. The elemental mercury removal at lower temperatures is favorable due to the exothermic nature of the adsorption process, and the interference by acid gases will be limited due to their low concentrations compared to the upstream treatment of flue gas.

2. Materials and methods

2.1. Materials

Coconut husk was obtained from T&H Coconut Fiber Sdn. Bhd. (Johor, Malaysia). For the synthesis and surface treatment procedures, acetic acid (CH_3COOH , 100.0%), hydrochloric acid (HCl, 97%), sodium hydroxide (NaOH, 99.0%), hydrogen peroxide (H_2O_2 , 30.0–32.0%), nitric acid (HNO_3 , 36.5–38.0%), sodium hypochlorite solution (NaOCl, activated chlorine < 10%) obtained from Merck (Germany), Sigma–Aldrich (USA) and Kanto Chemical Co. Inc. (Tokyo, Japan) were used as received. Deionized water produced by the Purite Water System (U.K) model Select Analyst HP40 was used to prepare all solutions. Nitrogen gas (purified, 99%) was obtained from Mega Mount Industrial Gases Sdn. Bhd. (Johor, Malaysia). The elemental mercury vapor was generated using a Metronics Dynacal permeation device (type HE-SR) purchased from VICI Metronics Inc. (USA).

2.2. Synthesis of adsorbents

2.2.1. Pristine

The coconut husk was mechanically separated into coconut pith (CP) and coconut fiber (CF) with a de-husking device. The CP and CF samples were washed several times using deionized water to remove dust and soluble impurities. The washed CP and CF were then oven dried at 50 ± 1 °C for 2 days, sieved between 70 and 100 μm particle sizes using standard sieves. These samples were denoted as CP-Pristine and CF-Pristine, respectively.

2.2.2. Surface treatments

Alkali treatment known as mercerization, was carried out according to the method proposed by Sreekala et al. (1997). The alkali treated samples (i.e. CF-NaOH or CP-NaOH) were prepared by agitating CP-Pristine or CF-Pristine with 5% NaOH solution using a temperature controlled shaker at constant speed of 200 rpm at 30 ± 1 °C. After 2 h, the mixture was washed using deionized water, followed by using water containing a few drops of acetic acid to neutralize the excess NaOH solution. Bleaching treatment (CP- H_2O_2 or CF- H_2O_2) was performed according to a method described by Brígida et al. (2010). Two grams of CP-Pristine or CF-Pristine were soaked with 40 mL of H_2O_2 basic solution prepared using 0.05 g of NaOH and 18 mL of H_2O_2 diluted to 100 mL using deionized water. The mixture was stirred at 200 rpm and 80 ± 1 °C for 2 h. The treated samples were then rinsed using deionised water. Both CP-NaOCl and CF-NaOCl samples were prepared by soaking 2 g of CP-Pristine and CF-Pristine respectively in 40 mL of NaOCl solution (v/v in water) for 2 h at 30 ± 1 °C.

All samples were initially dried at ambient temperature (30 ± 1 °C), followed by drying in an oven at 50 ± 0.1 °C for two days and then stored in desiccator.

2.3. Characterization

The changes in the surface morphology of adsorbent samples were determined by analyzing their respective SEM images, which were obtained using a scanning electron microscope (SEM) model JEOL JSM-6390LV (Japan). The surface chemistry of the adsorbent surfaces was analyzed by an FTIR (PerkinElmer Model 2000, USA). The FTIR spectra measurements were carried out over 4000–400 cm^{-1} using the KBr method. The determination of moisture and ash content of the samples was carried out according to E1755-01, (2015) and E1756-08, (2015), respectively.

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