MODIFICATIONS OF COCONUT PITH AS MERCURY ADSORBENTS FOR INDUSTRIAL APPLICATIONS

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy (Chemical Engineering)

Faculty of Chemical and Energy Engineering
Universiti Teknologi Malaysia

APRIL 2016

To my beloved family for their love and support

ACKNOWLEDGEMENT

Firstly, I praise to Allah, the Almighty, on whom ultimately we depend for sustenance, guidance and also for me giving me a strength and ability to complete this study.

I would like to express my special appreciation and thanks to my supervisor Assoc. Prof. Dr. Hanapi bin Mat for being a tremendous mentor, giving his continuous support and guidance of my Ph.D study. His patience, motivation and immersed knowledge help me in all the time of research and writing of this thesis. My sincere thanks are also to the Ministry of High Education, Malaysia for the financial support under MyBrain15 program. I would like to thank to the UTM's staffs and technicians for providing me with all the necessary facilities for the research.

I am thankful to my lab-mates at Advanced Materials and Process Engineering (AMPEN) Research Laboratory for the support, friendship and exciting learning environment. Thank you for all the fun we had during our study.

Last, but not least, very special appreciations to my parents and all my family members who inspired, encouraged and fully supported me for every trial that comes my way.

ABSTRACT

Contamination of wastewater by mercury ions poses a great concern due to its toxicity and threat to the public health and ecological systems. This study was conducted to investigate utilization of coconut pith (CP) as adsorbents for the removal of mercury ions, Hg(II) and MeHg(II). The CP underwent several modification processes: pre-treatment; silane-grafting and dye-loading, aiming for better Hg(II) and MeHg(II) adsorption performance. The adsorption performance study was conducted in batch and continuous adsorption system. The physical and chemical properties of CP adsorbents changed after modifications. The silane-grafting using mercaptopropyltriethoxysilane (CP-MPTES) and dye-loading using Reactive Red 120 (CP-RR) resulted in the highest removal efficiency towards both mercury ions. This is due to the presence of functional groups which have high affinity towards both mercury ions. Batch adsorption studies found that the adsorption capacity of both mercury ions was dependent on initial pH, adsorbent dosage, initial concentration, contact time and temperature. The maximum adsorption capacity of Hg(II) onto CP-Pure, CP-MPTES and CP-RR was 2.60, 2.61, and 2.60 mmol/g, while 0.50, 1.13 and 0.76 mmol/g was observed for MeHg(II), respectively. The equilibrium and kinetic data analyses found that the mechanism of mercury ions adsorption onto CP adsorbents is a combination of physical and chemical processes. The high regenerability was only observed in Hg(II) adsorption. The competence of Hg(II) and MeHg(II) adsorption in oilfield produced water and natural gas condensate samples, respectively, found that the presence of other metal ions reduced the adsorption performance of the mercury ions. The mercury ion adsorption in continuous fixed-bed adsorber studied at various conditions showed that the increase of flow rate and initial concentration caused the exhaustion time (t_{exh}) to occur earlier, but higher bed height prolonged the t_{exh}. The breakthrough curves of mercury ions adsorption were analyzed using Thomas, Bohart-Adam, Yoon-Nelson, Wolborska and Clark models. Thomas and Yoon-Nelson models fit most of the experimental data. However, empty bed contact time analysis found that the Yoon-Nelson model is more applicable to predict the breakthrough curves of the scale-up adsorber. The regenerability studies had low performance towards Hg(II) adsorption and fair performance towards MeHg(II) adsorption. In overall, the batch and continuous mercury ion adsorption results show the potential application of the CP adsorbents as low-cost adsorbent for industrial mercury ion removal process.

ABSTRAK

Pencemaran air sisa oleh ion raksa menimbulkan banyak kebimbangan disebabkan ketoksikannya dan ancaman terhadap kesihatan awam dan sistem ekologi. Kajian ini dijalankan untuk mengkaji penggunaan habuk kelapa (CP) sebagai penjerap untuk menyingkirkan ion-ion raksa, Hg(II) and MeHg(II). CP telah menjalani pelbagai proses pengubahsuaian: rawatan-pra, pencantuman-silana dan pemuatan-pewarna, bertujuan untuk mendapatkan prestasi penjerapan ion raksa yang lebih baik. Kajian prestasi penjerapan telah dijalankan dalam sistem penjerapan berkelompok dan berterusan. Ciri-ciri fizikal dan kimia penjerap CP berubah selepas pengubahsuaian. Pencantuman-silana menggunakan merkaptopropiltrietoksisilana (CP-MPTES) dan pemuatan-pewarna menggunakan Reaktif Merah 120 (CP-RR) menunjukkan keupayaan penyingkiran tertinggi terhadap kedua-dua ion raksa. Ini disebabkan kehadiran kumpulan berfungsi yang mempunyai kecenderungan yang tinggi terhadap ion raksa. Kajian penjerapan berkelompok menunjukkan keupayaan penjerapan ion raksa bergantung kepada pH awal, dos zat penjerap, kepekatan awal, masa sentuhan and suhu. Keupayaan maksimum penjerapan Hg(II) terhadap CP-*Pure*, CP-MPTES and CP-RR adalah masing-masing 2.60, 2.61 dan 2.60 mmol/g, manakala 0.50, 1.13 and 0.76 mmol/g terhadap MeHg(II). Analisa data keseimbangan dan kinetik menunjukkan bahawa mekanisma penjerapan ion raksa pada penjerap CP adalah gabungan proses fizikal dan kimia. Kajian penjanaan semula menunjukkan penyingkiran yang tinggi diperoleh hanya terhadap penjerapan Hg(II). Kecekapan penjerapan ion Hg(II) and MeHg(II) masing-masing pada sampel air hasilan lapangan minyak dan gas asli cecair menunjukkan kehadiran ion logam lain mengurangkan prestasi penjerapan ion raksa. Penjerapan ion raksa di dalam proses berterusan lapisan terpadat telah dikaji pada pelbagai keadaan menunjukkan peningkatan kadar aliran dan kepekatan awal menyebabkan masa penat (t_{exh}) terjadi lebih awal, tetapi lebih tinggi lapisan melambatkan lagi t_{exh}. Lengkuk bulus bagi penjerapan ion raksa dianalisa menggunakan model Thomas, Bohart-Adam, Yoon-Nelson, Wolborska dan Clark. Model Thomas dan Yoon-Nelson sesuai dengan kebanyakan data ujikaji. Walau bagaimanapun, analisa masa sentuhan lapisan kosong menunjukkan model Yoon-Nelson adalah lebih bersesuaian untuk meramal lengkuk bulus bagi penjerap berskala besar. Kajian penjanaan semula menunjukkan prestasi yang rendah terhadap penjerapan Hg(II) tetapi sederhana terhadap penjerapan MeHg(II). Secara keseluruhannya, keputusan kajian berkelompok dan berterusan terhadap penjerapan ion raksa menunjukkan potensi penggunaan zat penjerap CP sebagai penjerap yang murah untuk penyingkiran ion raksa industri.

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LIST OF SYMBOLS

A_c	-	Clark constant
A_T	-	Temkin isotherm constant (L/g)
B	-	Langmuir constant related adsorption energy (L/mol)
b_T	-	Temkin constant related to heat of adsorption (J/mol)
C_o	-	Initial concentration (mmol/L)
C_t	-	Concentration at time t (mmol/L)
D	-	Diameter of column/ adsorber
D_{eff}	-	Effective diffusivity (cm^2/min)
D_f	-	Film diffusion coefficient (cm^2/min)
D_M	-	Diffusivity of adsorbate molecule
D_p	-	Pore diffusivity (cm^2/min)
d_p	-	Particle diameter (m)
D_s		Surface diffusivity (cm^2/min)
E_a	-	Activation energy from Arrhenius (J/mol)
$E_{D\text{-}R}$	-	Activation energy from Dubinin-Radushkevich (kJ/mol)
F	-	Flow rate (mL/min)
h_{o1}	-	Initial adsorption rate from PFO model (mmol/g.min)
h_{o2}	-	Initial adsorption rate from PSO model (mmol/g.min)
k_1	-	Rate constant of PFO kinetic model (min^{-1})
k_2	-	Rate constant of PSO kinetic model (g/mmol.min)
K	-	Kelvin
K_b	-	Thermodynamic equilibrium constant (L/g)
k_{BA}	-	Rate constant of Bohart-Adam model (L/mmol min)
k_f	-	Freundlich constant relatedto adsorption capacity ($L^n mol^{1-n}$/g)
k_{fd}	-	Fickian's diffusion (min^{-1})
k_{TH}	-	Thomas rate constant (mL/mmol min)
k_{YN}	-	Yoon-Nelson constant (1/min)
M	-	Mass of adsorbent (g)
M_B	-	Molecular weight of water (g/mol)
N	-	Freundlich constant
N_o	-	Saturation concentration (mmol/L)
q_e	-	Amount adsorbed (mmol)
Q_e	-	Adsorption capacity at equilibrium (mmol/g)

Q_{e1}	-	Adsorption capacity from PFO (mmol/g)
Q_{e2}	-	Adsorption capacity from PSO (mmol/g)
$Q_{m.D-R}$	-	Adsorption capacity from Dubinin-Radushkevich model (mmol/g)
$Q_{m.L}$	-	Adsorption capacity from Langmuir model (mmol/g)
Q_o	-	Adsorption capacity of the virgin adsorbent (mmol/g)
Qr	-	Adsorption capacity of the regenerated column (mmol/g)
Q_t		Adsorption capacity of the regenerated column (mmol/g)
Q_{TH}	-	Adsorption capacity from Thomas model (mmol/g)
r_c	-	Clark constant
R_L	-	Dimensionless separation factor
R_p	-	Particle radius (cm)
S	-	Surface area per mass adsorbent (m^2/g)
T	-	Temperature (^oC)
t_b	-	Time at break point (min)
t_{exh}	-	Time at exhaustion point (min)
V_A	-	Liquid molar volume of adsorbate at boiling temperature
V_{bed}	-	Volume of column/ adsorber (m^3)
V_{eff}	-	Volume of effluent (L)
Z	-	Bed height (m)
Z_{min}	-	Minimum bed height
A	-	Initial adsorption rate of Elovich kinetic model (mmol/g.min)
B	-	Desorption constant from Elovich model (g/mmol).
β_{D-R}	-	Dubinin-Radushkevich constant (J/mol)
β_L	-	External mass transfer coefficient (cm/s)
β_{WB}	-	Kinetic coefficient of external mass transfer from Worborska model (min^{-1})
ε	-	Void fraction (dimensionless)
Δ	-	Film thickness (cm)
H	-	Removal efficiency (%)
μ_f	-	Fluid viscosity (Pa.s)
N	-	Linear flow rate (cm/min)
ρ_f	-	Fluid density (kg/m^3)
ρ_p	-	Particle density (g/cm^3)
T	-	Time required to achieve 50% breakthrough (min)
Y	-	Superficial velocity (m/min)
Φ	-	Associate parameter of water

ΔG^o	-	Gibbs free energy change (kJ/mol)
ΔH^o	-	Enthalpy change (kJ/mol)
ΔP	-	Pressure drop (Pa)
ΔS^o	-	Entropy change (kJ/mol)

LIST OF ABBREVIATIONS

AEPTES	-	Aminoethylaminopropyltriethoxysilane
APTES	-	Aminopropyltriethoxysilane
ARE	-	Average relative error
BTESPT	-	Bis(triethoxysilylpropyl) tetrasulfide
CF	-	Coconut fiber
CP	-	Coconut pith
CV-AAS	-	Cold vapor atomic absorption spectrometer
EBCT	-	Empty bed contact time
F-AAS	-	Flame atomic absorption spectrometer
FTIR	-	Fourier transform infrared
HETU	-	Height of an equivalent transfer unit
ICP-MS	-	Inductively coupled plasma mass spectrometer
MPS	-	Methacrylopropyltrimethoxysilane
MPSD	-	Marquardt's percent standard deviation
MPTES	-	Mercaptoproyltriethoxysilane
NGC	-	Natural gas condensate
OPW	-	Oilfield produced water
RE	-	Regeneration efficiency
SEM	-	Scanning electron microscopy
URS	-	Ureidopropyltriethoxysilane

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Agricultural based materials are lignocellulosic materials consisting of lignin, hemicellulose, cellulose and other organic matters (Rangabhashiyam *et al.,* 2013). These components have variety of oxygen functional groups (e.g. hydroxyl, carboxyl, phenol and ketone) that can act as active sites for adsorption of mercury ions. Other chemicals such as organosilanes, dyes, surfactants, and proteins can also be easily incorporated at these oxygen active sites. Therefore, agricultural based materials have gained a lot of interests as alternative adsorbent precursors.

Although variety agricultural based materials have been explored as potential adsorbent for targeting mercury ions, most of them have only focused on inorganic mercury, Hg(II) removal. Adsorbents such as desiccated coconut waste (Johari *et al.,* 2013), coconut fiber (Johari *et al.,* 2014b), rice straw (Song *et al.,* 2014), and *Carica papaya* wood (Basha *et al.,* 2009) show a high adsorption capacity towards Hg(II). However, they cannot guarantee to have a high adsorption capacity towards organic mercury ions such as methylmercury and phenylmercury. It is thus necessary to investigate the performance of these adsorbents towards other mercury species, since in most cases the waste effluents often contain other mercury species result from transformations that occur within the waste solutions (Lee *et al.,* 2016). A few studies also have been employed for the removal of both inorganic and organic mercury ions such as a LW-CHAR and LW-AC (Saman *et al.,* 2015), RPSR (Saman *et al.,* 2015), chitosan and modified-chitosan (Kushwaha *et al.,* 2010; Kushwaha and

Sudhakar, 2011), *Lemna minor* (Li *et al.,* 2011), *Streptococcus pyogenes* (Tuzen *et al.,* 2009) and *Coriandrum sativum* (Karunasagar *et al.,* 2005). Although, some of these adsorbents show good adsorption towards both mercury ions, it might be expensive to be used for small and medium scale industries.

This study was conducted to prepare new low cost adsorbents using coconut pith (CP) as the raw materials, aiming for high adsorption performance towards both inorganic, Hg(II) and organic methylmercury, MeHg(II). The CP was selected as raw material because it abundantly available. The CP was used widely as adsorbents for removing various heavy metals such as Cr(IV), Cd(II) and Ni(II) (Bhatnagar *et al.,* 2010). However, there has been no study conducted so far to investigate the comparative adsorption behavior of both organic and inorganic mercury ions. In order to enhance the mercury ions adsorption performance, the raw CP was modified using pre-treatment process, silane-grafting and dye-loading. The modifications provided additional Lewis base functional groups (i.e. oxygen, nitrogen and sulfur functional groups) that would be beneficial to form complexion with Lewis acid, mercury ions.

1.2 Problem Statement

The presence of mercury in the environment has become of great public concerns due to its toxicity in which poses a significant threat to the public health and ecological systems (Pillay *et al.,* 2013). In environment, mercury may exist in three different forms namely elemental mercury (Hg^{o}), organic mercury (e.g. methylmercury, MeHg) and inorganic mercury (e.g. $HgCl_2$). Organic mercury especially methylmercury is recognized as the most toxic mercury form to humans (Feng and Qiu, 2008). The inorganic mercury is less adsorbed by living organisms as compared to the elemental mercury and organic mercury. However, the deposited of inorganic mercury in soil and water is biologically easy to transform to more toxic organic mercury. It was reported that, the major risk of mercury released associated with organic mercury (i.e. methylmercury), because it tend to bioaccumulate in human food chain.

Among of various mercury removal processes, adsorption was more preferred due to its cheapness and high quality of treated effluents (Qadeer, 2007). Various materials have been evaluated as potential adsorbents for mercury removal such as silicas, carbons, zeolites, synthetic polymers, agricultural residues, biomass, peats, and industrial residues (Sharma *et al.,* 2015). Adsorption using carbonaceous materials was widely explored and the world consumption of carbon (especially activated carbon) is steadily increasing because of its effective application for treating various pollutants. However, despite its effectiveness, commercial activated carbon is expensive (Mohan *et al,* 2001; Pan *et al,* 2012), since the raw materials used for its preparation are normally from coal and timber, which are non-renewable resources. Therefore, persistent efforts have been made for developing highly effective, low-cost and locally available adsorbents (Rangabhashiyam *et al.,* 2013). There are many different studies on the use of low-cost materials such as agricultural wastes (Bhatnagar *et al.,* 2010; Demirbas, 2008; Kumar, 2006; Mallampati *et al.,* 2015), sewage sludge carbon (Zhang *et al.,* 2005), industrial wastes (Hovsepyan and Bonzongo, 2009; Tang *et al.,* 2015), peats and soils (Bulgariu *et al.,* 2008) for removing mercury.

The waste materials from agricultural industries are the common sources of low-cost adsorbents due to their abundance and less expensive as compared to other materials (Adegoke *et al.,* 2015). Many researchers have published and highlighted the use of agricultural residues as low-cost materials to adsorb heavy metals (Wan Ngah and Hanafiah, 2008; Bhatnagar *et al.,* 2010; Patel, 2012), dyes (Bhatnagar *et al.,* 2010; Patel, 2012; Rangabhashiyam *et al.,* 2013; Adegoke *et al.,* 2015), and other inorganic and organic pollutants (Bhatnagar *et al.,* 2010; Patel, 2012; Ahmad *et al.,* 2010). These agriculture residues have been utilized as adsorbents either in pristine or modified forms (Wan Ngah and Wan Hanafiah, 2008; Bhatnagar *et al.,* 2010; Patel, 2012; Rangabhashiyam *et al.,* 2013; Adegoke *et al.,* 2015; Ahmad *et al.,* 2010). The use of agricultural based materials as adsorbents would result in additional benefits to the humankind and environment since it could at least provide economic benefits, as well as solving waste disposal problems.

1.3 Objectives and Scopes

The objectives and scopes of the present study are:

1. To synthesize, modify and characterize the coconut pith as mercury adsorbents.

The CP-Pure was prepared from the raw coconut pith (CP) by grounding, sieving, washing with double-distilled water, and finally vacuum drying at 30±0.1 ^{o}C.The modification of the CP-Pure was carried out using several chemical modification methods which can be categorized into three processes: (a) pre-treatment; (b) silane-grafting and (a) dye-loading. The pre-treatment processes were conducted to remove soluble inorganic or organic substances that cover the outer layer of the CP. The pre-treatment agents namely sodium hydroxide (NaOH), sodium hypochlorite (NaOCl), hydrogen peroxide (H_2O_2), nitric acid (HNO_3), and laccase solution. The silane-grafting is a process of incorporation of organosilane into the CP-Pure. In the present study, the organosilanes namely mercaptoproyltriethoxysilane (MPTES), aminopropyltriethoxysilane (APTES), aminoethylaminopropyltriethoxysilane (AEPTES), bis(triethoxysilylpropyl) tetrasulfide (BTESPT), methacrylopropyltrimethoxysilane (MPS), and ureidopropyltriethoxysilane (URS) were used to functionalize the CP-Pure. The dye-loading is a process in which the dye reagent was incorporated into the CP-Pure. In the present study, the Reactive Black B (BB), Reactive Red 120 (RR), Methylene Blue (MB) and Methyl Orange (MO) were used to treat the CP-Pure.

After modifications, the modified CP adsorbents (or CP adsorbents) were characterized in order to understand their physical and chemical properties. The morphology of the CP adsorbents was analyzed using scanning electron microscope (SEM). The surface characteristics and chemical functional groups of the adsorbent were analyzed by determining their point zero charge (pH_{pzc}) and Fourier transform infrared (FTIR) spectra, respectively. The CP adsorbents were then evaluated for inorganic and organic mercury adsorption capacity carried out at standardized conditions using synthetic inorganic and organic mercury solutions prepared from the mercury nitrate monohydrate ($Hg(NO_3)_2.H_2O$) and methylmercury chloride (CH_3HgCl) salts, respectively. The CP adsorbents having high performance towards inorganic and organic mercury for each modification methods were selected for further batch and continuous adsorption studies.

2. To evaluate the adsorption performance of inorganic and organic mercury ions in batch adsorption system.

Adsorption of inorganic and organic mercury was carried out for the selected CP adsorbents having high adsorption capacity towards mercury ions. The adsorption capacity was analyzed at equilibrium and time dependent (kinetics) experiments. The adsorption mechanism was proposed based on the characterization and adsorption results obtained at various process parameters.

At equilibrium, the effect of initial solution pH, adsorbent loading, initial concentrations, and process temperature was studied. Adsorption at different pH values was conducted first in order to determine the optimum pH which was then selected to be used for other experiments. The adsorption isotherm data obtained at different mercury concentrations was analyzed by using the existing adsorption isotherm models namely Langmuir, Freundlich, Dubinin-Radushkevich (D-R) and Temkin models. Thermodynamic parameters such as the standard Gibbs free energy change (ΔG^o), standard enthalpy change (ΔH^o) and standard entropy changes (ΔS^o) were calculated from the temperature dependent adsorption experiments.

The adsorption kinetics was investigated from the adsorption time dependent experiments. The data then were further analyzed by using the existing kinetic models: (a) physical and (b) chemical reaction based kinetic models. The physical models of diffusion process used to determine external mass transfer rate, film diffusion coefficient and internal pore diffusion coefficient. The chemical reaction models including pseudo-first order (PFO), pseudo-second order (PSO) and Elovich models were used to interpret the chemical step process.

In order to investigate the potential applications of the CP adsorbents, the regeneration and selectivity studies were carried out. The regeneration study was carried out by adsorption- desorption process activities. The solutions of 0.1 M of HCl and KI were used as eluents or regeneration agents. The competence of Hg(II) and MeHg(II) adsorption with metal ions were analyzed using the oilfield produced water (OPW) and natural gas condensate (NGC), respectively.

3. To evaluate the adsorption performance of inorganic and organic mercury ions adsorption in continuous adsorption system.

The continuous adsorption process was evaluated using fixed-bed adsorber. The adsorption performance was described through the concept of breakthrough concentration curves. The effect of bed height, initial mercury concentration and flow rate was investigated. In order to understand the adsorption characteristics of the fixed-bed adsorption, the cumulative adsorption data was analyzed using isotherm and kinetic models. The existing isotherm models namely Langmuir, Freundlich, Dubinin-Radushkevich (D-R) and Temkin isotherm models were used to analyze the adsorption isotherm data. The pseudo-first order (PFO), pseudo-second order (PSO), Elovich and Fickian's kinetic models were used for the adsorption kinetic data analysis. The breakthrough curves of mercury ions adsorption were analyzed using Bohart-Adam, Thomas, Yoon-Nelson, Wolborska, and Clark models.

The selected CP adsorbents having good adsorption performance were selected for further adsorption design and scale-up studies. Lastly, the regenerability studies of CP adsorbents towards Hg(II) and MeHg(II) was carried up using the selected regeneration agents. The adsorption performance of the regenerated adsorbent were studied up to 4 desorption cycles.

1.4 Thesis Outline

This thesis has five chapters. Chapter 1 describes briefly of research background, objectives and scopes of the study. A critical review on mercury in the environment, potential adsorbents for mercury adsorption and technical aspects of mercury adsorption system and operation are presented in Chapter 2. Chapter 3 presents the overall methodologies used which include (i) the materials used, (ii) the adsorbent preparations and modification procedures, (iii) adsorbent characterization procedures, and (iv) adsorption process procedure in batch and continuous system. The findings of the study were presented in Chapter 4. In this chapter, the effect of modifications towards inorganic and organic mercury ion adsorption was discussed. Several factors affecting the adsorption performance towards both mercury ions were evaluated in batch and continuous adsorption systems. Summary of research findings and the recommendations for the future works are presented in Chapter 5.

1.5 Summary

The existence of organic and inorganic mercury even at low concentration can cause severe impacts to the human health as well as to the environment. The removal of mercury through adsorption process has nowadays been widely adopted. The success of this process is required for the development of new adsorbents that are cheap and selective towards mercury. In this study, the coconut pith (CP) was used as a raw material (precursor) for the development of low-cost mercury adsorbents. The CP was modified through various methods towards obtaining good CP adsorbents for both organic and inorganic mercury ions. The mercury adsorption performance of CP adsorbents was carried out in the batch and continuous adsorption systems. Several parameters were investigated in order to understand the mercury adsorption mechanisms.

5.3 Concluding Remarks

The low-cost coconut pith (CP) based adsorbents were synthesized through various modification methods for removal inorganic and organic mercury from synthetic aqueous mercury solution and wastewaters carried out in batch and continuous adsorption systems. The modifications successfully improved the adsorption capacity and removal efficiency of mercury ions especially for MeHg(II) removal. Depending on the interplay of adsorbent characterizations and adsorption performances at various conditions (e.g. pH, process temperature, isotherm and contact time), diverse mercury-binding mechanisms can take places during the adsorption processes (e.g. physical and chemical interactions), as well as physical process mechanisms (e.g. film and surface diffusion). In addition, the continuous adsorption processes provide a practical adsorption application in real industrial processes. The modelling approach of the adsorption performance in the continuous process can be used for a scale-up adsorber data. Thus, the findings of this research could be useful for better understanding of the coconut pith as mercury ions adsorbents towards developing new inexpensive adsorbents.

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