

CHARACTERIZATION OF OIL PALM EMPTY FRUIT BUNCH AND RICE HUSK BIOCHARS AND THEIR POTENTIAL TO ADSORB ARSENIC AND CADMIUM

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ABSTRACT

Biochar as a new soil amendment has a potential in controlling the fate of trace elements in the soil system. However, the production of biochar from different feedstocks and pyrolysis conditions resulted in variable biochar properties which have an influence on trace elements availability. The experimental works are focused on physical and chemical properties of biochars produced from Empty Fruit Bunch (EFB) and Rice Husk (RH) as heavy metals adsorbents. The morphology of EFB biochar comprise rigid structure with uniform pores size. The surface area of biochars ranged between 23.22-46.32 m² g⁻¹, dominated with mesopores. The chemical characterization of biochars revealed high carbon content in EFB biochar compared to RH biochar (54.08 and 7.78%). Both biochars are alkaline in nature (>pH 9) and contain substantial amounts of N, P, K, Ca and Mg. Fourier transform infra-red spectra showed the heterogenic functional groups on EFB biochar surface and domination of silica content in RH biochar. The batch experiment was employed to determine the adsorption capacity of these biochars for As and Cd. The adsorption data were fitted well in Langmuir isotherm with high correlation coefficient (R²>0.9) for As and Cd. The q_{max} of EFB biochar for As and Cd was higher than RH biochar. The q_{max} of EFB biochar was 0.4240 and 15.1515 mg g⁻¹ for As and Cd, respectively. In contrast, the q_{max} of RH biochar for As and Cd was only 0.3522 and 3.1908 mg g⁻¹. The results show that EFB biochar and RH biochar have potentials as good sorbent for As and Cd. Therefore, it can be suggested that the application of these biochars may possibly reduce the trace elements availability in the contaminated soil.

Keywords: Morphological Study, FTIR, Chemical Characterization, Adsorption Study, Langmuir Isotherm

1. INTRODUCTION

Biochar is gaining attention from people worldwide due to its potential for soil conditioning as well as for the improvement of the soil physico-chemical properties. The high organic carbon content in biochar can increase carbon storage in the soil system when applied to the soil. The presence of complex functional groups in biochar, as a stable organic matter is highly recalcitrant against the

decomposition. Therefore, biochar has a potential to enhance long-term carbon pool storage in the soil system (Lehmann *et al.*, 2003).

Soil organic matter is known to have major influence on soil properties and controls the toxicity, transport, fate and behavior of non-polar organic compounds in soils. It reduces the metal (loid)s mobility by redistributing the trace elements to less-available forms (Shuman, 1999). Biochar is believed to have potential in remediating the contaminated soil by adsorption and complexation

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reactions on trace elements (Harter and Naidu, 1995; Bradl, 2004). Biochar is a stronger sorbent than other forms of organic matter with the very high affinity and capacity for sorbing organic compounds (Yang and Sheng, 2003). Sorption to biochar also have been reported less reversible than other organic matter (Braidia *et al.*, 2003; Sander and Pignatello, 2007). A number of studies have reported the addition of biochar to soil have influenced the heavy metals mobility and bioavailability (Cheng *et al.*, 2006; Park *et al.*, 2011; Jiang *et al.*, 2012; Samsuri *et al.*, 2013). Namgay *et al.* (2010) reported that the addition of wood biochar decreased the concentration of As, Cd and Cu in maize shoots. Rongjun *et al.* (2014) also reported the reduction in soil extractable Cd and uptake by rice plants grown in contaminated rice paddy with the application of wheat straw-derived biochar. The potential of biochar in reducing the trace elements and contaminants availability is attributed to its physico-chemical properties such as porosity, surface area, pH, cation exchange capacity and organic functional groups.

Biochar from many sources of biomass can be produced through the pyrolysis process in the absence of oxygen. Different types of biochar have different characteristics depending on the feedstock and pyrolysis conditions. In Malaysia, there are two potential biomasses from industrial wastes that can be used as biochar; oil palm Empty Fruit Bunches (EFB) and Rice Husks (RH). It is reported that around 20 million tonnes of EFB (MPOB, 2012) and 0.41 million tonnes of RH (DAM, 2012) was produced annually during the processing in the mills. The high availability of these feedstocks, can be used as good sources for the biochar production. The applications of these biomasses as biosorbent in removing environmental pollutants in soil and aqueous solution also have been reported previously (Mahvi *et al.*, 2004; Chowdhury *et al.*, 2011; Hussaro, 2014). In addition, the conversion of EFB and RH biomasses to biochar can be an alternative to the sustainable management of the industrial waste. However, there is very limited information on the properties of commercial EFB biochar and RH biochar produced in Malaysia to be used as soil amendments in remediating contaminated soil. Therefore, this study was conducted to investigate the physico-chemical properties of EFB biochar and RH biochar responsible for immobilizing trace elements in the soil system.

2. MATERIALS AND METHODS

2.1. Sample Preparation

Empty fruit bunch biochar was collected from Nasmeh Technology Sdn Bhd located in Dengkil in

collaboration with Universiti Putra Malaysia. The biochar was produced from the pyrolysis of oil palm empty fruit bunch in a pilot plant carbonator applied with medium pyrolysis temperature (300-350°C). Meanwhile, the RH biochar was collected from BERNAS (Malaysian National Rice Agency) rice mill in Tanjung Karang, Selangor which was produced at pyrolysis temperature of approximately 500°C. The biochars were air dried and characterized in triplicates based on physical and chemical properties.

2.2. Biochar Analysis

The pH (H₂O) and Electrical Conductivity (EC) of biochar was measured in a 1:2.5 (v:v) and 1:5 (v:v) biochar: Water suspension, respectively. The biochar total carbon was analyzed using LECO CR-412 carbon analyser (LECO Corporation, St. Joseph, MI). The total nitrogen (Bremner, 1996), total P, total K and CEC (ammonium acetate, pH 7, leaching method) of biochar samples were determined using autoanalyzer (QuickChem FIA 8000 Series, Milwaukee, Wisconsin). The concentrations of Ca and Mg of biochar were determined by using the dry ashing method (Baker *et al.*, 1964) with atomic absorption spectrophotometer AAnalyst 400 (Perkin Elmer, Norwalk, CT, USA). Total As and Cd content in biochar was determined using atomic absorption spectrophotometer after digestion with aqua-regia (1:3 HCl to HNO₃) solution. The surface morphology of biochar was obtained through JSM-6400 Scanning Electron Microscope (SEM, JEOL USA, Peabody, MA) at Institute of Bioscience, Universiti Putra Malaysia, Serdang, Malaysia. The Brunauer, Emmett and Teller (BET) surface area, pore size and surface area distributions, micro-pore volume and surface area of biochar were measured with AUTOSORB-1 (Quantachrome Instruments, Boynton Beach, Florida, USA). The organic functional groups of biochar were studied by using the Perkin Elmer Spectrum 100 Fourier Transform Infra-red Spectrometer (Perkin Elmer Corporation, Norwalk, CT, USA).

2.3. Adsorption Study

Sorption of As and Cd on biochar was measured by using the batch method. An amount of 2 g subsamples of biochar were placed in each of the eight centrifuge tubes and equilibrated with 0.01 M CaCl₂ solution containing a series of Cd concentrations (0, 20, 40, 60, 80, 100, 150 and 200 mg L⁻¹) by adding CdCl₂ solution. In the case of As, sodium arsenate (HAsNa₂O₄·7H₂O) solution of similar concentration

was added to the biochar. Each sorption set, both for As and Cd, were replicated twice. The biochar mixture was equilibrated overnight (16 h) at room temperature on a rotary shaker at 30 revolutions per minute (rpm). Then the suspension was centrifuged at 3000 rpm for 15 min followed by filtration through Whatman No. 1 filter paper. The concentrations of As and Cd in the clear extract solution was determined using the Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES, Perkin Elmer, Optima 8300, USA). Amount of As and Cd adsorbed by the biochar can be calculated from the difference between the initial and final concentration of these metals in the equilibrium solution. The data was then fitted to the Langmuir equation.

2.4. Statistical Analysis

Means comparison of chemical properties between EFB and RH biochar were performed using T-test comparison with JMP statistical software, Version 9.0.1 (SAS Institute Inc., Cary, NC, USA).

3. RESULTS

3.1. Chemical Characterization of Biochar

The elemental composition for EFB biochar and RH biochar are listed in **Table 1**. Both biochars have alkaline pHs (>pH 9) with pH value of oil palm EFB biochar slightly lower than RH biochar. The EC values for EFB biochar and RH biochar were 5.33 and 2.90 dS m⁻¹, respectively. The empty fruit bunch biochar had almost five times higher total carbon content and CEC value as the RH biochar. Both biochars were found to contain substantial amounts of nutrients with high concentration of potassium (K). Biochars also have low concentration of arsenic and cadmium ranging from 1.15 to 0.45 mg kg⁻¹.

3.2. Morphology of Biochar

Figure 1 shows the Scanning Electron Microscope (SEM) images of the two biochars. The EFB biochar possessed uniform pores and smooth wall surfaces under 1000x magnification. In comparison, the pores on RH biochar are not well shaped with diminished structure of pores. Small pores also were detected on the rough surface of RH biochar. From the images, it can be seen that the pores on both biochars have similar size with maximal diameter of 10 µm.

3.3. BET Surface Area

Surface area and porosity of EFB biochar and RH biochar are presented in **Table 2**. Biochar produced from

EFB had a larger surface area (46.32 m² g⁻¹) than RH biochar (23.22 m² g⁻¹). Another important feature of biochar as adsorbents is the size and distribution of pores which can be determined through the nitrogen adsorption as well. The micropore volume of EFB biochar was found to be the same as RH biochar (≈0.01 cm³ g⁻¹). Internal surface area of biochar which represents pore on the inner wall as a result of interior crack was referred to as micropore area. Meanwhile, the average pore diameter for both biochars are in the range of mesopores diameters, with the internal pore width between 2 and 50 nm.

3.4. Functional Groups of Biochar

Figure 2 shows FTIR spectra for the EFB biochar and RH biochar from the FTIR spectroscopy. Two adsorption peaks were observed at 3365.43 and 2924.69 cm⁻¹ corresponding to the O-H and C-H group stretching, respectively, attributable to the cellulose component of EFB biochar (Abdul Khalil *et al.*, 2011). The adsorption peak as observed at 1374.47 cm⁻¹ is associated with the bending vibration of stronger C-H functional group. A strong peak at 1572.94 cm⁻¹ is due to the aromatic C = C stretching. The peak at 1007.92 cm⁻¹ represents the C-O stretching due to the presence of phenols and alcohols. Meanwhile the adsorption peaks between 1000 and 650 cm⁻¹ indicate the presence of alkenes group. Unlike the FTIR spectrum shown by EFB biochar, the RH biochar spectrum illustrates less adsorption peaks with wavelength of less than 1300 cm⁻¹. The sample only showed strong peak intensity at 1050.89 and 788.65 cm⁻¹ which indicate the presence of Si-O stretching.

3.5. Adsorption Isotherm

The adsorption isotherm data were fitted to the Langmuir's adsorption model. **Table 3** shows the values of adsorption isotherm parameters for EFB biochar and RH biochar. The maximum adsorption capacity (q_{max}) of EFB biochar for As was 0.424 mg g⁻¹, which is higher than RH biochar (0.352 mg g⁻¹). Similar trend was found on q_{max} of Cd with the values of 15.15 and 3.19 mg g⁻¹, for EFB biochar and RH biochar, respectively. The parameter b is related to the affinity of the binding sites, which allows comparisons of the affinity of biochar toward the metal (loid) ions. RH biochar had a higher binding affinity (b) for As and Cd than did EFB biochar. Value of R² shows correlation or linear relationship, whereas the relationship become more linear when the value is closer to 1. The high correlation coefficient values (R²) which ranged from 0.98 to 0.99 indicate that the Langmuir isotherm best fitted the experimental data.

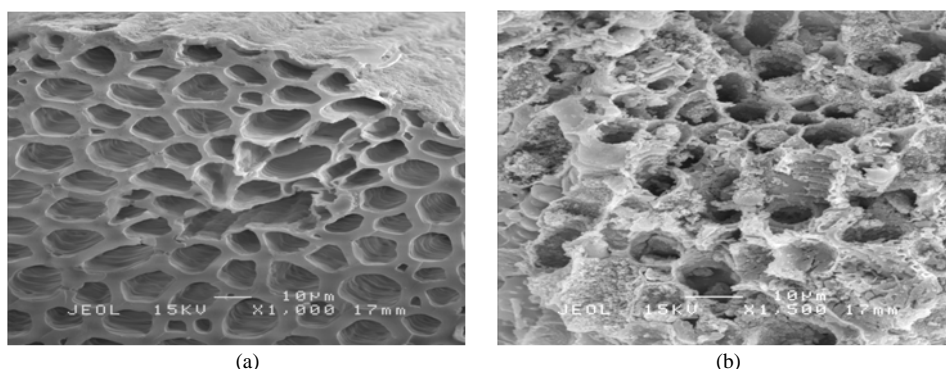


Fig. 1. SEM image of (a) EFB biochar and (b) RH biochar

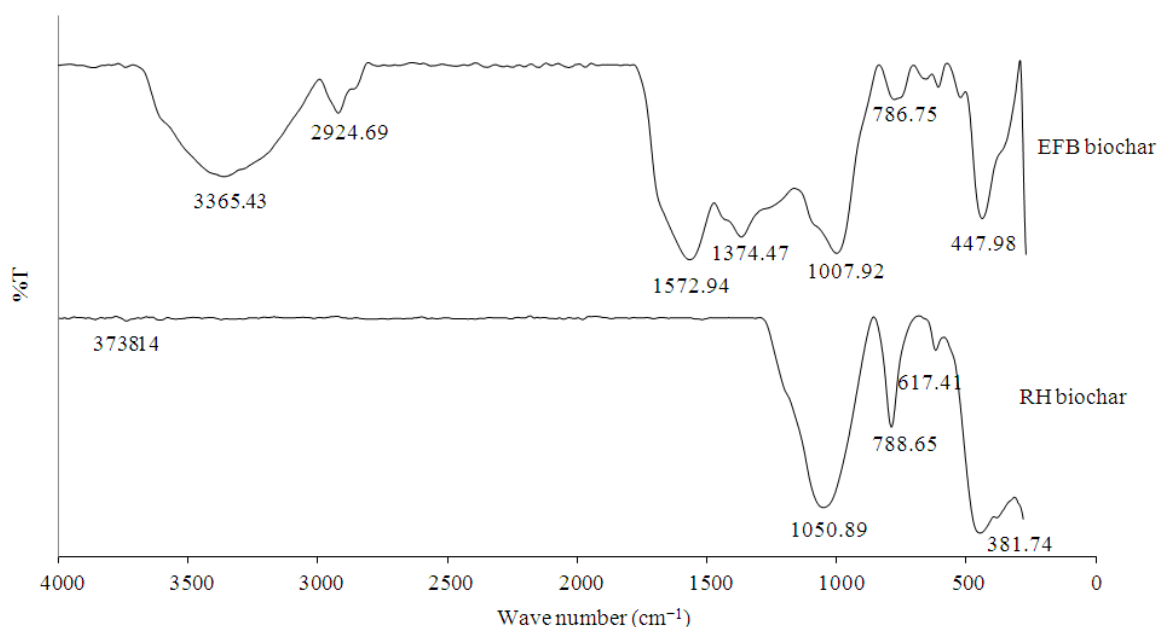


Fig. 2. FTIR spectra for EFB biochar and RH biochar. Wave numbers of interest are labelled

Table 1. The chemical properties of EFB biochar and RH biochar

	EFB biochar	RH biochar
pH (1:2.5 H ₂ O)	9.47 a	10.24 b
EC (dS m ⁻¹)	5.33 a	2.90 b
Total carbon (%)	54.08 a	7.78 b
CEC (cmol _c kg ⁻¹)	63.93 a	13.45 b
Total nitrogen (%)	1.63 a	0.23 b
Phosphorus (%)	0.21 a	0.36 b
Potassium (%)	5.3 a	0.72 b
Calcium (%)	0.11 a	0.02 b
Magnesium (%)	0.13 a	0.08 b
Arsenic (mg kg ⁻¹)	1.15 a	0.55 a
Cadmium (mg kg ⁻¹)	0.80 a	0.45 b

Note: Values with different letters within the row are significantly different at p<0.05, t-test comparison

Table 2. BET surface area and porosity of biochar

Biochar	Surface area (m ² g ⁻¹)	Micropore volume (cm ³ g ⁻¹)	Internal surface area (m ² g ⁻¹)	Average pore diameter (nm)
EFB	46.32	0.008	0.61	3.85
RH	23.22	0.009	1.41	4.34

Table 3. Sorption isotherm obtained by fitting the data with the langmuir isotherms for the efb biochar and rh biochar

Biochar	Heavy metal	Langmuir model		
		q _{max} (mg g ⁻¹)	b (L mg ⁻¹)	R ²
EFB	As	0.4240	0.0073	0.9890
	Cd	15.1515	0.1142	0.9921
RH	As	0.3522	0.0248	0.9823
	Cd	3.1908	0.6920	0.9984

4. DISCUSSION

Empty Fruit Bunch (EFB) and Rice Husk (RH) are two affordable and sustainable feedstocks in Malaysia to be made as biochar. Most studies were investigating the effects of biochars from different feedstock on soil fertility and plant growth performance with less information on trace elements interaction. The potential of biochar as a sorptive media depends on its physical and chemical properties. The physical properties of biochars are mainly affected by pyrolysis condition, especially, temperature (Boateng, 2007). When high temperature (500°C) was applied during the production of RH biochar, the pore size enlarged and the wall between adjacent pores were destroyed, which explained the diminishing pore structure of RH biochar (Zhang *et al.*, 2004). The presence of inorganic material from the feedstock ash also was observed on RH biochar surface which can be partially filling or blocking the porosity (Shinogi and Kanri, 2003; Lee *et al.*, 2010). This phenomenon is verified by BET analyses that showed the lower BET surface area of RH biochar compared to EFB biochar. However, BET surface areas for both biochars are comparable to the biochars examined in the literature ranging from 1.89 to 141 m² g⁻¹ (Mohamad *et al.*, 2011; Anchan and Kunio, 2012; Samsuri *et al.*, 2013). Generally, both biochars exhibit mesopores diameter (≈4 nm) which indicate the potential of adsorption capacity of biochars at the liquid-solid interface (Bagreev *et al.*, 2001). Indeed, this have been confirmed by sorption experiment results for As and Cd through Langmuir isotherm (**Table 3**).

The variation in chemical properties of biochar derived from EFB and RH (**Table 1**) are due to differences of the original biomass and their production process (Sukiran *et al.*, 2014). Rice husk was heated at high temperature for energy production in the rice mill, thus producing many mineral ashes which resulted in higher pH than EFB biochar. Biochars from pyrolysis process usually are alkaline in nature with pH of more than 7.00 (Cheng *et al.*, 2006; Namgay *et al.*, 2010; Van Zwieten *et al.*, 2010) attributed from ash residues, consistent with the result of present study. The low total carbon content of RH biochar is attributed to the high temperature as well, with greater loss of C. In contrast, the production of EFB biochar by using carbonator, under lower temperature and controlled process, resulted in minimal loss of C with high value of CEC. This finding agrees with work of Leng *et al.* (2011) who found high total C content of pineapple residue biochar produced at lower temperature. The lower temperature in producing

EFB biochar also resulted in significantly higher level of EC, which suggests the higher retention of mineral salts from the process. This can be explained from for the higher nutrients concentration, except for phosphorus, found in EFB biochar compared to RH biochar.

The regression correlation coefficients for As and Cd are very high according to the Langmuir equation (**Table 3**), with R² value closest to 1. The adsorption capacity, q_{max} shows the higher sorption of As and Cd by EFB biochar than RH biochar. The adsorption maximum capacity according to the Langmuir isotherm in this study for Cd was higher than As for both biochars. Mohan *et al.* (2007) reported the same trend of adsorption capacity (Cd>As) for pine wood char, oak wood char, pine bark char and oak bark char. The pH and CEC are two important factors that attributed to sorption mechanism of trace elements with addition of biochar (Kumpiene *et al.*, 2008). The alkaline properties of biochars increased the solution pH, which induced metal immobilization through metal precipitation and decreases metal solubility (Rees *et al.*, 2013). Samsuri *et al.* (2013) reported that the increased in As (III) adsorption increased with the increasing pH for EFB biochar and RH biochar with maximum adsorption at pH 8. Meanwhile, the amount of As(V) adsorbed by these sorbents increased with the increasing of pH between pH 2 and pH 6 and decreased after pH 7.

The rise in soil pH resulted from the application of biochar also enhanced metal sorption with the increase of the negative charge density on biochar surfaces (Harvey *et al.*, 2011). The formation of functional groups especially carboxylic-C and aromatic OH during oxidation (Liang *et al.*, 2006) possibly led to the formation of biochar-trace elements complexes on biochar surfaces. Han *et al.* (2013) reported that the removal of Cd²⁺ from aqueous solution by rice straw biochar may be attributed to the formation of surface complexes between Cd and carboxyl or hydroxyl groups. The high stability of this complexes are recalcitrant to the environment, promising for long-term soil remediation, when applied to the soil.

5. CONCLUSION

The physico-chemical properties of biochar derived from EFB and RH feedstocks showed potential as adsorbents to reduce As and Cd mobility. The porous structure with high alkaline and adsorption sites of these biochars can influence As and Cd mobilization by sorption and complexation mechanism. However, the biochar properties can vary depending on the type of feedstocks

and pyrolysis conditions. Therefore, study on the pyrolysis of EFB and RH produced at different temperature is highly recommended to evaluate the effect on biochar properties as adsorbents. The field experiments to assess the effect of these biochars on the trace elements availability in soil and uptake by plant are required.

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