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Adsorption Behaviour of Selected Biochar for Cadmium in Black Soldier Fly (*Hermetia Illucens*) Larvae Fed On Contaminated Organic Waste

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ABSTRACT

The efficiency of rice husk biochar (RHB) and coconut shell biochar (CSB) in adsorbing Cadmium (Cd), as one of the prevalent heavy metals reported to bioaccumulate highly at the larval stage of the black soldier fly (BSF) was investigated. The synthesized biochars were activated using sulphuric acid and surface morphology investigated using SEM. The surfaces of CS biochar had well defined pores while RH had irregular pores of different sizes. Batch adsorption studies were done to optimize the effect of different adsorption parameters in three replicates. The equilibrium data was then used to design adsorption isotherms where Freundlich isotherm model best fitted both RHB and CSB data with R^2 values of 0.9967 and 0.9877 respectively while kinetic data was best described by pseudo second order model suggesting chemisorption process took place for both the adsorbents. The adsorption capacity of CSB and RHB were reported as 0.8484 mg×g^{-1} and 0.8399 mg×g^{-1} respectively. The efficiency of Cd removal by RHB and CSB was found to be 97% and 96% respectively. These results confirmed the suitability of RHB and CSB as adsorbents for effective removal of Cd in organic wastes.

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Introduction

Adsorption is widely used as effective physical method of separation in order to eliminate or lower the concentration of pollutants (organics and inorganics) in the polluted waters by application of most common adsorbents, such as silica gel, activated carbon, and aluminium oxide [1]. Biochar, as a potential adsorbent material, is a product of thermal decomposition of organic material under the limited supply of oxygen (O_2) at temperatures between 350 and 700 °C [1]. Cellulose rich biomass waste from agriculture and forestry (such as plant residues, wood waste, peat, cattle manure and others) is used as a feedstock [2]. Due to the results of various studies in applications of biochar and such characteristics as porosity, high specific surface area, cation exchange capacity, it would be perspective to develop biochar as an adsorbent material that can be efficiently used in remediation of heavy metal pollution in feedstocks [1, 3].

However, this great potential is challenged by the diversity of contaminants present in wastes. Due to poor disposal, other types of waste like e-waste, phosphate fertilizers, polyvinylchloride plastics, paints and batteries in which cadmium is used as stabilizer are often mixed with organic waste [4]. Safety concerns on the quality of larvae raised from such substrate to be used in the food system as feed ingredients have been raised [5, 6]. The major contaminants of concern in BSF farming using waste are chemical contaminants such as heavy metals since microbes are inactivated during innate detoxification processes [7, 8].

Heavy metals are not degradable and the continuous increase in heavy metal contamination is a cause for concerns as these metals have the ability to bioaccumulate in tissues of various biotas and may also affect the growth and development of various organisms [9]. In different organisms' compartments, absorption of metals across cell walls involves mostly soluble metal ions [10].

Recent studies have reported accumulation of heavy metals in BSF with cadmium metal accumulating at levels higher than the initial concentration levels in the feed [5, 6, 11, 12]. While there is growing body of research assessing the heavy metal accumulation in BSF larvae little is known about remediation strategies to minimize this risk. The constant monitoring of heavy metal content in the feed is technically unattainable by small holder farmers. This study therefore explored the potential of locally produced biochars from selected agricultural wastes to adsorb cadmium from feed waste, thereby, presenting a low-cost adsorbent that can effectively remediate the heavy metal accumulation in BSF larvae.

Materials and Methods

Production of Biochar

The agricultural waste used as biomass were rice husks (RH) and coconut shells (CS) collected from Ahero in Kisumu County and Malindi in Kilifi County respectively in Kenya. The equipment used for pyrolysis in this study was Top Lid Updraft Drum (TLUD) technology under limited oxygen conditions at a temperature of around 450 °C to 550 °C. This technology was preferred for its efficiency to pyrolyze feedstock without much smoke released to the atmosphere and its inexpensive design over sophisticated machines [13]. In this work, this equipment was constructed using a 200 L metallic drum, perforated at the base as an air intake section, a piece of iron sheet for chimney which was fitted on the drum lid and two iron bars. The TLUD was filled with selected biomass, one type at a time and fire was started at the top of the material. Once flames were well established, the drum was covered to reduce oxygen supply and biochar started forming under limited oxygen. Combustion started with white smoke and there after it was entirely "clean combustion" (no smoke).

The process was closely observed by the intensity of the flames which eventually became low while tossing some water at the sides of the drum. The tossing of water was used to indicate that the biochar was ready at instant puffs of steam as well as to prevent ash formation by maintaining the temperature. Pyrolysis process took 2 hours and 1.5 hours for rice husks and coconut shells respectively, thereafter biochar was cooled by sprinkling water as described by Van et al. and sun dried before crushing them and labelling them according to the feedstock parent material used [13].

Activation of Biochar

Prior to adsorption studies, both CSB and RHB were activated in an acid to enhance its efficacy. Biochar was ground using an electric grinder into smaller particle size for ease of use and incorporation into the substrate. Then biochar was later chemically activated by soaking using 1 molar sulfuric acid for 24 hours. Thereafter, the samples were washed with distilled water to remove the excess acid from the surface before oven drying them at 150 °C. The oven dried biochar was packed in air tight containers for adsorption experiments.

Characterization of Rice Husks and Coconut Shells Biochar

The surface morphology of the selected biochars was investigated using scanning electron microscope (SEM) (SEM: JEOL JSM-7500F) at the National Centre for Nanostructured Materials, Council for Scientific and Industrial Research (CSIR) in South Africa. The biochar samples were selected and carefully mounted on aluminum studs using conductive glue. Preconditioning was done by coating with a thin layer of carbon thereafter analyzed in a vacuum chamber, then the whole area of the biochar was scanned and photographed for analysis.

Batch Adsorption Studies

The capacity of CSB and RHB to adsorb cadmium at different parameters including biochar dosage, solution pH, contact time, Cd concentration and temperature was investigated. The adsorbents CSB and RHB were agitated at optimum times of 60 minutes and 30 minutes respectively in 50 mL of 1 ppm Cd standard solution. The pH adjustments were made using either 1 M of NaOH or 5 M of H_2SO_4. The effect of initial concentration on adsorption capacity of the two selected biochar types was monitored by varying cadmium concentrations from 0.25 to 3 ppm keeping other parameters constant. This was conducted at the recorded optimum contact time for each biochar. A magnetic mechanical shaker was used to stir the mixtures for the required time before filtration.

All adsorption experiments were performed in replicates under same conditions. The mixtures of biochar and the Cd solution were shaken at 30 rpm and removed at respective times and thereafter, biochar residues for both biochar type at all parameters were separated from the filtrates using Whatman filter paper No.1. Concentrations of the filtrates was determined using the flame atomic absorption spectrophotometer (AAS).

Equilibrium adsorption q_e (mg/g), was calculated from the mass balance equation 1:

$$qe = (C_o - C_e)/m \times v \quad (1)$$

Where C_o is the initial concentration in (ppm), C_e is the Cd concentration at equilibrium (ppm), V is the solution volume (L) and m is the mass of biochar dosage (g).

The percentage of cadmium removal was calculated as in equation (2) below;

$$\text{Removal } (\%) = (C_o - C_t)/C_o \times 100 \quad (2)$$

where, C_o and C_t are the initial and the final Cd concentration (mg/L) at time t, respectively.

Results and Discussion

Scanning Electron Microscope (SEM) Analysis

The morphological features of RHB and CSB were examined using SEM and the micrographs of the respective biochars at X5000 magnifications as shown in Figure 1. From these results, it was observed that the surface of RHB (Fig. 1a) had plenty of clear irregular crystals which covered the surface while a hollow nature of uneven pattern was observed from CSB (Fig. 1b) image. The roughness and coarse irregular crevices of distinct dimensions observed in both biochar types, indicated the presence of unevenly distributed different sized pores which are imminent adsorption sites. These cavities also serve as channels of adsorption giving access to the active adsorption sites of meso and micropores to interact with the adsorbate thereby allowing for more adsorption [14].

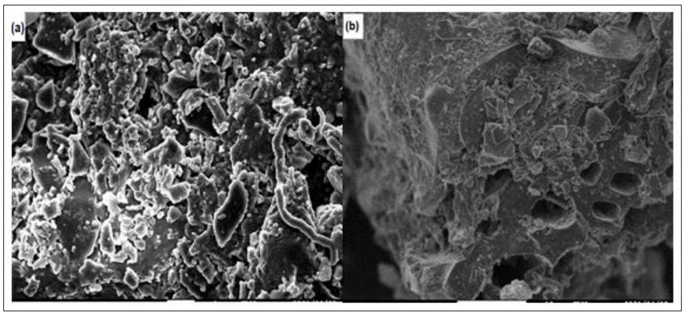


Figure 1: SEM Images for (a) Rice Husks Biochar and (b) Coconut Shells Biochar

Despite being subjected to same pyrolytic conditions, CSB developed a well-defined pore structure whereas RHB showed more irregular pore structure with different sizes and shapes. This could be due to lignin richness content of the biomass material. According to Mahdi et al., highly lignified feedstocks tend to produce macroporous biochars than feedstocks rich in cellulose which produce microporous [15]. In this case, coconut shells were more lignified than rice husks thus one reason for the variation. Moreover, previous studies attributed increased porosity of biochar to formation of internal pores as biomass volatilize during pyrolysis [16]. However, both biochars portrayed a heterogeneous distribution of pores and rough texture which is in agreement with Freundlich isotherm observed under kinetics studies of this work.

Effect of Contact Time

The adsorption of cadmium onto RHB and CSB was monitored at varying time intervals of 5, 10, 20, 30, 60 and 120 mins, keeping other parameters constant. cadmium was rapidly adsorbed onto RHB and CSB in the first 30 minutes as observed in Fig. 2a. The CSB showed a relatively low increase in the subsequent adsorption of Cd before reaching equilibrium at 60 minutes while RHB showed a slight reduction before levelling up. This trend was ascribed to a two-phase adsorption phenomena that is the prominently rapid phase and the relatively low phase which is due to the abundance of active adsorption sites on adsorbent surface which gradually become saturated with time [17-19].

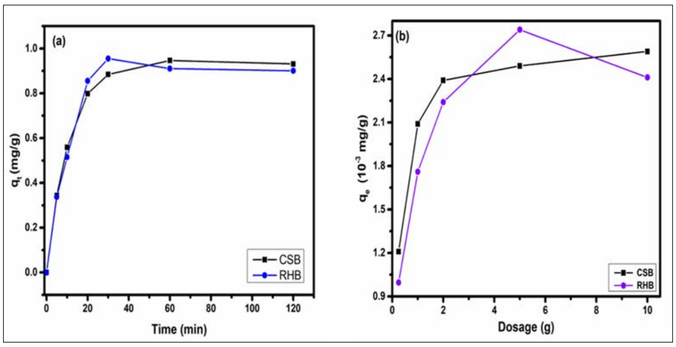


Figure 2: (a) Effect of contact time and (b) adsorbent dosage on Cd adsorption capacity of Cd

However, a slight decrease in adsorption shown by RHB from 30 minutes before saturation was attributed to desorption of weakly bounded Cd ions as adsorption sites on the adsorbent decreases. Similar results were reported for pine sawdust biochar on Cu (II) ions [18]. In this study, RHB was more effective in adsorption of cadmium than CSB because it took lesser time. Despite uniform experimental conditions that both adsorbents were subjected to, RHB and CSB reached adsorption equilibrium at different times of 30 and 60 minutes respectively. This meant that CSB needed longer time to reach equilibrium adsorption while RHB took a shorter time. The variation in attainment of equilibrium of the two biochars produced under same pyrolytic conditions was attributed to the difference in pore structure as RHB showed a more porous structure than CSB.

Effect of Adsorbent Dosage

The effect of biochar dosage on cadmium adsorption was studied by agitating different amounts of respective biochar (0.25, 1, 2, 5 and 10 g) in 50 mL of Cd standard solution. The effect of RHB and CSB dosages on adsorption of cadmium is shown in Figure 2b. cadmium adsorption was reported to increase with increase in dosage of the respective adsorbents. This was attributed to an increase in the total number of adsorption sites present on the surface of the adsorbents which, in turn, increases the overall binding of cadmium ions. While this remained true for rice husks biochar, coconut shells biochar only increased adsorption capacity up to 2 g before a slight reduction of approximately 0.001 and thereafter remained constant. This could mean that there were no more cadmium ions in the working standard that extra adsorbent material could bind. The metal ions in the solution were less than the exchangeable sites on the biochar thus less cadmium uptake [20]. Considering the SEM micrographs of the two adsorbents, RHB showed numerous irregular pores in comparison with CSB. Therefore, increasing the dosage directly increased adsorption sites thus the reason for continued increase in adsorption of RHB.

Effect of pH

The effect of pH on Cd removal was investigated on varying solution pH at 1.5, 3, 6, 7 and 12. The adsorption of Cd ions on RHB and CSB was studied over a pH range of 1.5 to 12 and the results illustrated in Figure 3a. Adsorption of Cd was relatively low at lower pH values of 1.5 to 2 but a rapid increase in Cd ions removal was observed when pH increased from pH 3. However, increasing pH to 12 yielded no significant changes for both biochar types due to possible reduction of the adsorption sites at basic pH. The Cd removal by CSB and RHB at acidic pH (less than 2) was low but increasing pH from 3 to 6 increased the rate of adsorption before reaching equilibrium. This was as a result of high competition between metal ions and hydrogen ions for the adsorbent sites that occurs at low pH thus decreased overall removal of the contaminant as similarly reported [21].

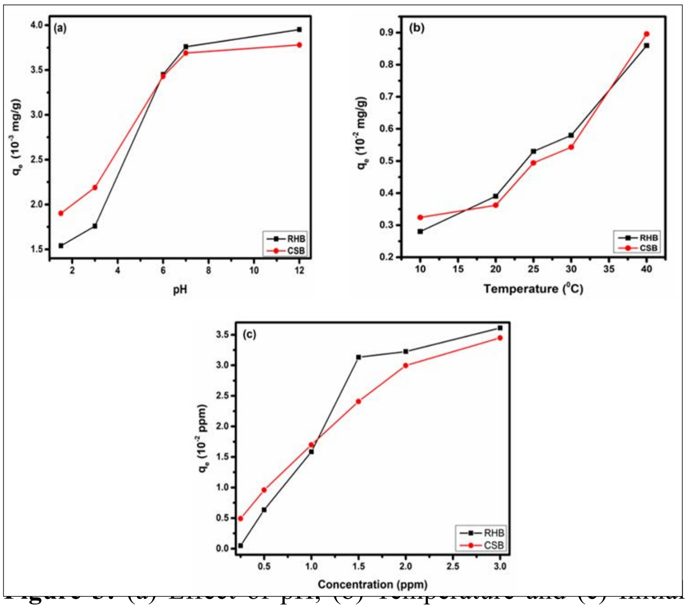


Figure 3: (a) Effect of pH, (b) Temperature and (c) Initial Concentration on Adsorption of Cd

The rapid adsorption observed with increasing pH from 3 was due to reduced competition between metal ions and hydrogen ions for the sorbent sites, as deprotonation of functional groups of the adsorbents discharged additional binding sites resulting in higher adsorption of the adsorbate [16]. The figure showed a reduction in the rate of adsorption as pH values approached 12, this entails that at higher pH, adsorption was less pronounced due to precipitation, which leads to formation of hydroxide complexes whose solubility is much lower [22].

Effect of Adsorption Temperature

The effect of solution temperature was investigated at intervals of 10 °C, 20 °C, 25 °C, 30 °C and 40 °C. Temperature is another parameter that affects adsorption capacity and kinetics rate of any adsorbent. Thus, adsorption capacity of cadmium on CSB and RHB was determined as a function of temperature. The sorption capacities of both biochars increased with increasing temperature, which is an indication of endothermic type of adsorption process as presented in Figure 3b. While both biochar types portrayed an endothermic adsorption process, the best performance on CSB and RHB was observed at 30 °C and 40 °C respectively. Taghlidabad et al. [23] reported similar results where the optimum temperature for best performance on the used biochars was 40 °C.

Effect of Initial Concentration

cadmium adsorption onto RH and CS biochar increased with increasing Cd concentration, thus the adsorption capacity of the total metal ion increased from the lowest concentration as shown in Figure 3c. This behavior was attributed to an increase in the mass transfer driving force which is a result of increasing number of collisions between the sorbent and the metal ions as the concentration increases [20]. Therefore, at optimum conditions, it could be concluded that the highest cadmium adsorption occurred at the highest initial metal concentration which was 3 ppm. These results concur with previous work of Gebretsadik et al., [24].

Adsorption Isotherms

The adsorption isotherms for the removal of cadmium by the two types of biochar was investigated using the Langmuir and Freundlich isotherm models. Adsorption models were constructed using cadmium initial concentration variation data at equilibrium points. Generally, the Langmuir model describes a monolayer adsorption of the adsorbate onto the localized adsorption sites. It assumes energies of adsorption as uniform with no transmigration of the sorbate in the plane of the surface [25] .The linearized form of Langmuir isotherm is expressed as

$$1/qe = 1/K_L q_{max} .\ 1/c_e + 1/q_{max} \ \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (3)$$

Where q_{max} represents that maximum adsorption capacity (mg/g) and K_L (L/mg) is the Langmuir's isotherm constant which shows the binding bond between cadmium and biochar.

The dimensionless Langmuir constant also known as the separation factor (RL) was calculated using equation 4

$$R_L = 1/1 + c_i \times K_L \ \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (4)$$

Where Ci is the initial metal concentration and RL is the Langmuir constant which indicate the adsorption possibility as favorable if it is ($0 < R_L > 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$).

Equation 5 represents the linearized form of Freundlich isotherm model which describes adsorption processes that is non-ideal and reversible. The model applies to multilayer adsorption with non-uniform distribution of energies and affinities over heterogeneous surfaces.

$$\log Q_e = \log_{kf} + 1/n \log_{Ce} \ \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (5)$$

Where k_f is the Freundlich's constant and used to measure the adsorption capacity and 1/n is the adsorption intensity. The value of 1/n demonstrates the adsorption process either favorable or unfavorable if 0.1< 1/n< 0.5 and 1/n> 2 respectively.

Table 1: Langmuir and Freundlich Isotherm Parameters for Cd Adsorption onto CSB and RHB

Langmuir					Freundlich		
Biochar	q_{max}	k_l	R_l	R^2	1/n	K_f	R^2
RHB	0.0023	4640.43	95.9547	0.9205	0.4882	0.0052	0.9967
CSB	0.0225	28.2924	12.1191	0.9222	0.3444	0.0347	0.9877

From these results and based on the experimental data for cadmium adsorption onto RHB and CSB, Freundlich model best fitted the adsorption process for both biochar types as shown in Figure 4. The isotherm parameters for both models are shown in Table 1. These parameters together with the linear regression values indicated that Freundlich model best fits the data, better than Langmuir model.

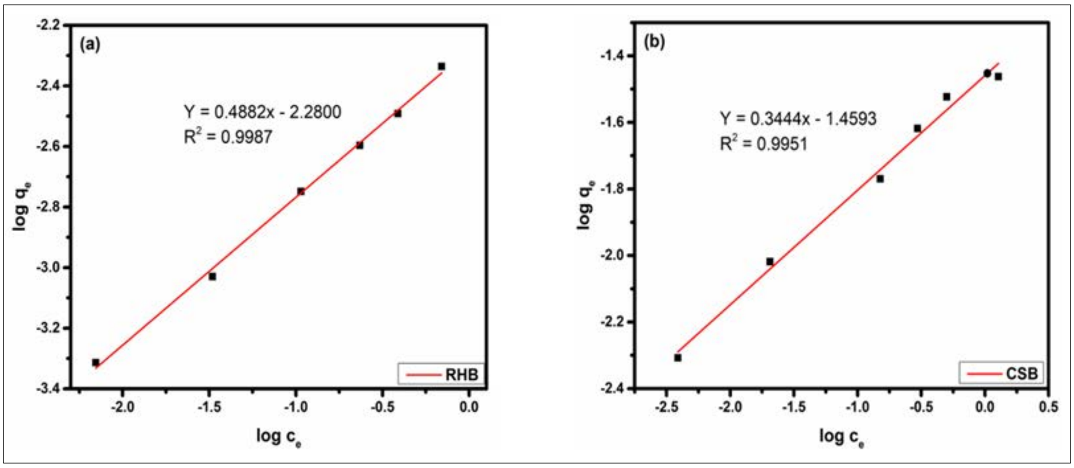


Figure 4: (a) Rice husks and (b) Coconut shell biochar adsorption isotherm models

Furthermore, the 1/n value which is the heterogeneity factor of CSB and RHB, was in the range 0 and 1 which is also an indication of favorable adsorption process [13]. This trend was also reported by Taghlidabad et al. [23] for biochar produced from apple and grape pruning residues. Puglla et al. also reported similar findings for cadmium and Lead onto peanut shells, Chonta pulp and corn cob biochar [26].

Previously, Doumer et al. described a similar trend for biochar produced from five different feedstocks [27]. Despite that, these results were opposite of other previous biochar adsorption studies which supported Langmuir isotherm model. Khan et al. used rice husks biochar on cadmium from soils with different water conditions and their results fitted on Langmuir model [28]. In this case the variation to our rice husks findings could be attributed to pyrolysis condition activation and experimental conditions. This therefore informs that biochar from different feedstocks produced at different temperatures have different adsorption capacities and mechanisms with regard to sorbate of interest [13]. In this study, cadmium adsorption on rice husks and coconut shells biochar was best described by the Freundlich model, which implied that adsorption process occurred on a heterogeneous multilayered surface.

Adsorption Kinetics

Furthermore, adsorption kinetics was investigated on both CSB and RHB using pseudo-first order (PFO) and pseudo-second order (PSO) models. The values of linear regression coefficients (R2) obtained from Equation 6 (PFO) and 7 (PSO) were used to predict the most suitable kinetic model for the process,

$\ln (q_e - q_t) = \ln q_e - k_1 t$……………………………………. (6)

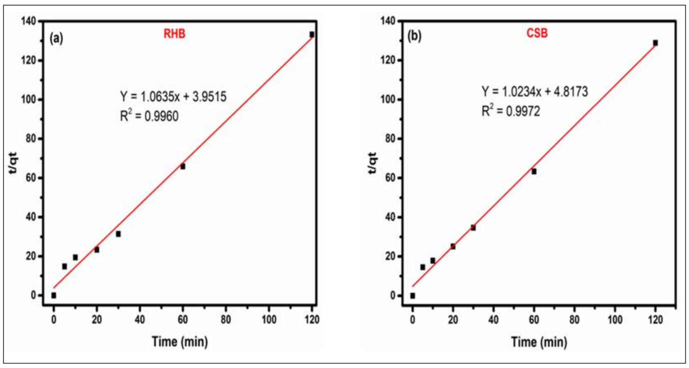
$t/q_t = 1/k_2 q_e^2 + t/q_e$… ……………………………..….... (7)

Where q_e is the amount of Cd adsorbed at equilibrium (mg/g), and q_t is the amount of Cd adsorbed at time t (mg/g). k_1 (min^{-1}) and k2 (g mg-1min^{-1}) are the equilibrium rate constants.

Table 2: Data for linear PFO and PSO models of adsorption kinetics

Order of reaction	Parameters	RHB	CSB
	$q_{, exp}$.(mg/g)	0.8345	0.8461
Pseudo 1st order	$q_{e, cal}$.(mg/g)	0.3926	0.4391
	k^1 (min^{-1})	-0.0010	-0.0004
	R2	0.5441	0.8102
Pseudo 2nd order	$q_{e, cal}$.(mg/g)	0.8399	0.8484
	k^2 (g $mg^{-1}min^{-1}$)	0.1857	0.1232
	R^2	0.9904	0.9934

The adsorption kinetic parameters of cadmium on CSB and RSB was further evaluated using Pseudo 1st order and Pseudo 2nd Order equations. The results of the fitting of both kinetic models to the cadmium adsorbed are shown in Table 2. Pseudo second order kinetics was observed to be the best fitted model in explaining the kinetics of cadmium adsorption on both biochars as shown in Figure 5.



This model showed higher value of linear regression coefficient (R^2) of 0.9904 and 0.9934 for RHB and CSB respectively when compared to Pseudo first order model with respective R2 values of 0.5441 and 0.8102. Furthermore, pseudo second order model was also shown by the closeness of the calculated adsorption capacity (q_e) and the experimental values (q_e). Based on these results, adsorption of cadmium on coconut shells and rice husks biochars occurred through chemisorption mechanism which is controlled by chemical processes like valence forces sharing or exchanging electrons between adsorbent and adsorbate and electrostatics [20].

Conclusions

The adsorption of Cd on CSB and RHB was found to be influenced by pH, biochar dosage, contact time, temperature and metal initial concentration. The results from this study showed that biochars prepared from agricultural wastes such as rice husks and coconut shells using the TLUD technology can effectively remove Cd from waste effluents. Freundlich isotherm model best fitted the adsorption isotherm and suggested that adsorption took place on heterogeneous surfaces. The SEM images depicted defined surface morphology in each biochar and also supported the occurrence of adsorption on heterogeneous surfaces. The efficiency of Cd removal by RHB and CSB was found to be 97 % and 96 % respectively. It is worth noting that RHB and CSB though considered less valuable can serve as feedstock materials with high adsorption capacity to adsorb heavy metals from livestock feeds for feed and food safety.

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