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TREATMENT OF ACID MINE DRAINAGE VIA ADSORPTION WITH FOOD WASTE ACTIVATED CARBONS

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Abstract: This study addresses the challenges of acid mine drainages (AMDs) treatment by exploring food waste-derived activated carbons (ACs). The study involves a synthesis of ACs from fruit peels and evaluation for removing heavy metals in AMDs. The AMD solutions were simulated and experiments were conducted via batch adsorption. All the ACs exhibited high adsorption affinities for Pb(II) in the order of KOH-AC > NaOH-AC > H_3PO_4-AC > H_2SO_4-AC > $ZnCl_2$-AC, however, the overall best adsorption capacities were recorded by KOH-AC, NaOH-AC and $ZnCl_2$-AC, with estimated equilibrium uptakes of 526.31±50.52, 26.14±0.23, 426.62±20.68, 164.24±11.54, 129.95±6.31 and 141.75±26.01 mg/g, for Hg(II), As(IV), Pb(II), Cd(II), Cu(II) and Co(II), respectively. The adsorption kinetics was faster for the smaller size metals than the larger ones. The study highlights the potential of these ACs as sustainable and effective adsorbents for mitigating the impact of mining activities on water quality. The findings may contribute valuably to the field of environmental remediation and sustainable mining.

Keywords: *acid mine drainage, activated carbon, adsorption, heavy metal, wastewater treatment*

1. INTRODUCTION

Toxic heavy metal pollution from the mining and hi-tech industries is among several environmental problems facing the world today (Akcil and Koldas, 2006; Acheampong, 2013; Bediako et al., 2017). Mining contributes immensely to the economy of many countries across the globe; however, mining activities also lead to the generation of acid mine drainage (AMD), which is always laden with numerous toxic heavy metals, such as As, Hg, Pb, Cd, Cu, Co, Ni, Cr, Al, Fe, etc. (Akcil and

Koldas, 2006; Oyewo et al., 2018). Having been recognized as one of the most severe environmental problems in the mining industry, the causes, predictions, and treatments of AMD have become the focus of many research initiatives commissioned by governments, the mining industry, universities, and research establishments, with additional input from the general public and environmental groups (Akcil and Koldas, 2006).

In many mineral-rich developing countries, the mining activities, most of which are illegally operated, have contaminated the surrounding environments and water bodies. A typical example is the case of "galamsey", also known as "gather and sell" in Ghana, which is currently causing an existential threat to human lives, as well as the fauna and flora, due to the mass pollution of major water bodies and agricultural farmlands. These issues are seen to emanate from either outright neglect of safety and mine wastewater treatment protocols or direct ejection of the toxic metal-bearing wastewaters into the surrounding environments without prior proper treatments (Acheampong, 2013; Bansah et al. 2016). The mining activities of both artisanal and legal miners are reported to endanger the environment through land degradation, deforestation, biodiversity loss, and contamination of surface and groundwater resources. The use of chemicals, commonly heavy metals and cyanide is of major concern. For example, the mercury amalgamation technique of gold extraction, which is largely practiced by small-scale miners in some parts of Africa, is harmful to a wide range of ecological entities (Bansah et al. 2016).

Heavy metal toxicity can cause serious carcinogenic and other adverse health problems even at low concentrations (Bediako et al., 2017). This is because they are easily accumulated by aquatic organisms, which can be passed on to human beings via the food chain; hence they must be properly treated (Li et al., 2015; Oyewo et al., 2018). Due to their severe toxicities and evolving health threats, international and domestic regulatory bodies have been revising and tightening regulations on their handling and treatments before discharge into the aquatic environment (Bediako et al., 2015; Khalil et al., 2013). Despite the strict regulations, some mining entities and illegal operators still manage to flout these directives. For instance, several surveys conducted by the Environmental Protection Agency (EPA) of Ghana showed that not even the major companies adhered to the permissible discharge levels of heavy metal-bearing AMD wastewaters into water bodies (Acheampong, 2013; Ghana EPA, 2010). Lack of adequate human resource capacities to manage wastewater or the absence of low-cost wastewater treatment options are identified as the two main reasons for the non-compliance by the companies (Acheampong, 2013).

The conventional methods of AMD wastewater treatment such as cementation and chemical precipitation techniques are expensive and pose a tendency of secondary pollution through sludge formation. Conversely, adsorption has been identified as one of the best alternative treatment methods owing to its simplicity, low-cost, environmental sustainability, and efficiency for low concentrations up to parts per billion (Bediako et al., 2016; Li et al., 2013). Moreover, there are several classes of adsorbents to choose from, and adsorption using activated carbon (AC) is one of the most widely known options. However, a major issue confronting the patronage of AC is the high costs of commercial products, which are usually sourced from coal, coir, and petroleum residues (Yagsi, 2004; Bediako et al., 2018). Recent studies have suggested the use of food waste-derived ACs as alternatives, which have received considerable acceptance owing to the ready availability and abundance of food waste precursors, such as fruit peels, shells, nuts, and seeds. Therefore, in this study, fruit peels were used as precursors to synthesize low-cost ACs and to subsequently evaluate their efficiency in heavy metal removal from potential AMDs. The study offers important perspectives for advancing environmental remediation and fostering sustainable mining practices through effective treatment of mining wastewater.

2. MATERIALS AND METHODS

2.1. Chemicals used

Metal complexes including $HNa_2AsO_4.7H_2O$, $Pb(NO_3)_2$, $Cd(NO_3)_2.4H_2O$, $Cu(NO_3)_2.3H_2O$, $Co(NO_3)_2.6H_2O$, and $HgCl_2$, and chemical reagents such as H_2SO_4, KOH, NaOH, H_3PO_4, $ZnCl_2$ and HCl, were procured from Daejung Chemicals and Metals Co., Ltd. and Sigma-Aldrich Ltd. All the chemicals used in this study are of analytical grade and were used as received, without further treatment. Stock solutions (~1000 mg/L) were prepared from the metal complexes and diluted into working solutions of 0–1000 mg/L for the adsorption experiments.

2.2. Activated carbon synthesis method

Dry pulverized orange peels were first pre-carbonized at 400 ºC for 1 h. After that, they were soaked in the activation agents, i.e., KOH, NaOH, H_2SO_4, H_3PO_4, and $ZnCl_2$ in 3:1 weight ratio for 24 h, followed by activation at 800 ºC for 1 h under continuous N_2 purging. In all, five types of activation agent-based ACs were produced. These ACs were washed with distilled water (DW) and dried in an oven kept at 70 ºC for 24 h, pending adsorption evaluation. The synthesized ACs were characterized as previously reported and as discussed later (Bediako et al., 2018; Bediako et al., 2020).

2.3. Adsorption studies

The adsorption studies were performed in batch modes using the prepared metal solutions of As(V), Hg(II), Pb(II), Cd(II), Cu(II), and Co(II) as representatives of the heavy metals that usually accompany AMDs. First, each of the five ACs was evaluated against each of the six heavy metal ions. After that, the best AC for each of the metal species was selected to run adsorption isotherm studies at pH 6 with initial concentrations ranging from 0–1000 mg/L. Approximately 10 mg of each AC sample was weighed into a 50 mL falcon tube and 20 mL of each metal solution was added. The mixtures were placed in a multi-shaking incubator whirling at 120 rpm and 25±2 ºC for 24 h. After equilibrium, the solutions were centrifuged at 12000 rpm for 5 min and diluted for analysis of the residual concentrations of metal ions.

Next, adsorption kinetics were carried out with multi-metal mixtures of all the metal ions. About 300 mL of 100 mg/L metal solutions were filled into glass beakers covered with aluminum foils and placed on multi-purpose magnetic stirrers with stirring bars. AC dozes of 0.1 g were then added to the solutions and timers were set to start counting. Small portions (~ 1 mL) were drawn from the bulk of each solution at various time intervals for 24 h and then analyzed for residual concentrations. An inductively coupled plasma-atomic emission spectrometer (Thermo Scientific, iCAP 7000 series, ICP Spectrometer, USA) was used for the analyses, and the uptakes were calculated according to the expression:

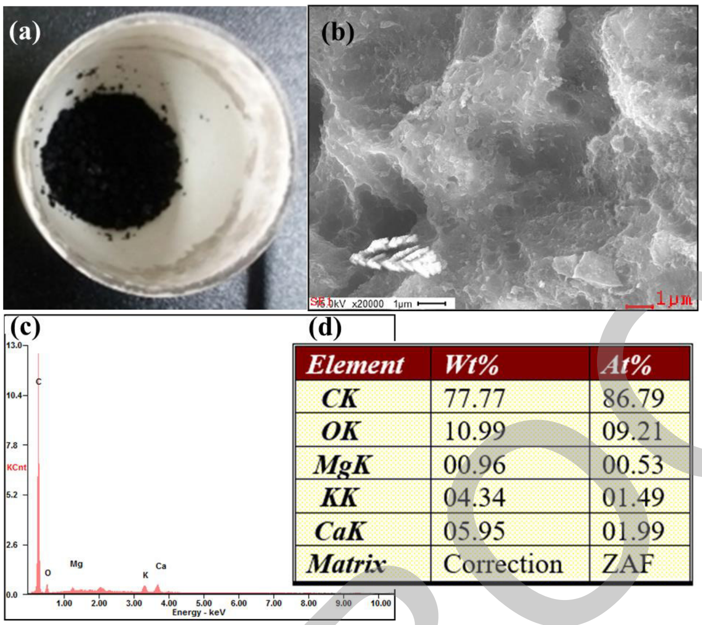
$$q = \frac{(C_i V_i - C_e V_e)}{M} \tag{1}$$

where C_i and C_e are the initial and equilibrium metal ion concentrations in mg/L, V_i and V_e are the initial and equilibrium volumes in L, and M is the dry weight of ACs in grams.

3. RESULTS AND DISCUSSION

3.1. Characterization of the synthesized ACs

The synthesized ACs were examined physically by means of digital camera, as well as using a field emission scanning electron microscope (FE-SEM, SUPRA 40VP), manufactured in Germany by Carl Zeiss. This equipment was integrated with an energy dispersive X-ray spectroscope (EDX) and could provide information on both the surface morphologies and elemental constitution of the ACs. Using the KOH-AC as a representative, Figure 1a presents a digital image of the synthesized ACs, showing clear formation of black granulated substances that are symbolic of typical ACs. The FE-SEM image captured at x20000 magnification portrays varying porosities of the ACs at an average size of 1 μm (Figure 1b). Consequently, evidence of elements including C, O, Mg, K, and Ca is present in the ACs, according to the EDX spectrum in Figure 1c. As expected, the carbon content is the highest at ~78 wt% and ~87 At% (Figure 1d).



Element	Wt%	At%
CK	77.77	86.79
OK	10.99	09.21
MgK	00.96	00.53
KK	04.34	01.49
CaK	05.95	01.99
Matrix	Correction	ZAF

Figure 1

KOH-AC showing formation of black granules typical of traditional ACs in (a) a digital camera, (b) FE-SEM image showing surface morphology, (c) and (d) EDX results showing elemental spectra and table of element composition, respectively

3.2. Evaluation of heavy metal removal capacities

Initial adsorption tests were conducted to evaluate the adsorption affinity of each of the synthesized ACs towards the heavy metal ions, and to select the best adsorbent for each metal ion. The metal uptakes by the ACs using 100 mg/L initial concentrations of each metal in a multicomponent system are plotted in Figure 2. As can be seen, the adsorption capacities differed according to the types of activation agents used. The As(V) and Co(II) uptakes had been relatively low for all the ACs; however, the $ZnCl_2$-AC and NaOH-AC showed differences by recording significantly high uptakes of 15.30 and 38.58 mg/g, respectively for the aforementioned metals. The best adsorbents for Pb(II) and Hg(II) were identified to be the KOH-AC and $ZnCl_2$-AC, which exhibited uptakes reaching 210.17 and 194.32 mg/g, respectively. The Cd(II) and Cu(II) appeared to be best adsorbed by the H_3PO_4-AC, which recorded uptakes of 151.37 and 161.96 mg/g, respectively; however, careful observations of the supernatants revealed that large amounts of precipitations had occurred. For this reason, the next best adsorbent, i.e., NaOH-AC was considered for the subsequent adsorption tests for these two metals. Overall, all the ACs showed high adsorption capacities of Pb(II) due to its large size since the uptake was calculated on a milligram per gram basis. A summary of the selected best ACs and their respective heavy metal uptakes are given in Table 1.

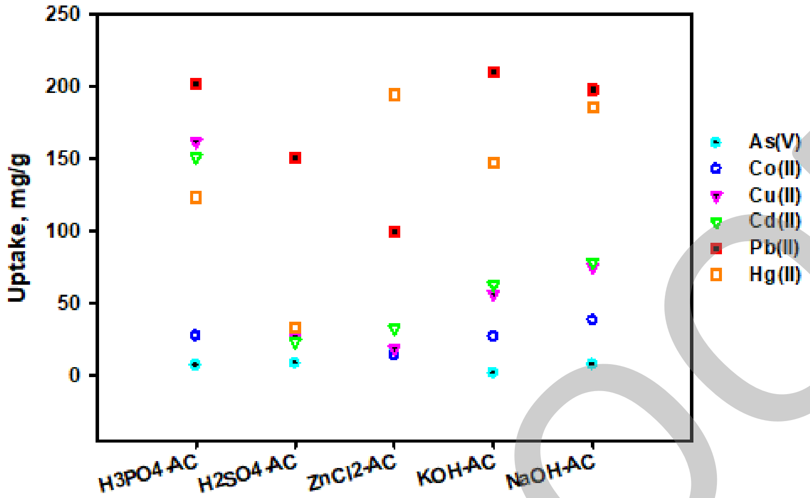


Figure 2

Activation agent-based carbons and their heavy metal uptakes. Experimental conditions: 10 mg AC in 20 mL of 100 mg/L multi-metal solution at pH 6, 120 rpm, and 25±2 °C for 24 h

Table 1

Summary of best AC adsorbents for the representative heavy metals

Adsorbent	Metal specie	Uptake (mg/g)
$ZnCl_2$-AC	As(V)	15.30
NaOH-AC	Co(II)	38.58
NaOH-AC	Cu(II)	75.49
NaOH-AC	Cd(II)	78.65
KOH-AC	Pb(II)	210.17
$ZnCl_2$-AC	Hg(II)	194.32

3.3. Adsorption isotherm evaluation

Following Table 1, adsorption isotherm studies were performed using the selected best ACs for each of the respective target heavy metals. The isotherm experiments were carried out to obtain the maximum equilibrium uptake of each metal by the ACs. It could be observed from Figure 3 that all the uptakes followed the usual adsorption phenomenon where the adsorption capacity increases but removal efficiency decreases comparatively with increasing initial concentrations (Abudaia et al., 2013; Bediako et al., 2016). In other words, the isotherm data points rose steadily and reached near plateau-like shapes at equilibrium. Each data was fitted with the two most utilized isotherm models; the Langmuir and Freundlich models, to estimate the maximum equilibrium uptakes and intensities of interactions. These models are presented in the following equations:

I) Langmuir model:

$$q_e = q_m \frac{bC_e}{1+bC_e} \tag{2}$$

II) Freundlich model:

$$q_e = K{C_e}^{1/n} \tag{3}$$

where q_m is the maximum equilibrium uptake given in mg/g, b is the coefficient concerning the affinity between the ACs and heavy metal ions, q_e is the equilibrium uptake (mg/g) at any time, C_e is the equilibrium metal ion concentration (mg/L), K and n are the Freundlich constants depicting the relative adsorption capacity and adsorption intensity, respectively.

The Langmuir model provided better fits to the isotherm data with high values of determination coefficient (R^2) than the Freundlich model (Tables 2 and 3). Moreover, the Langmuir model's predicted order of maximum equilibrium uptakes was consistent with that from the experiments. The order was $ZnCl_2$-AC-Hg > KOH-AC-Pb > NaOH-AC-Cd > NaOH-AC-Co > NaOH-AC-Cu > $ZnCl_2$-AC-As. Although the $ZnCl_2$-AC recorded the highest adsorption capacity of 526.31±50.52 mg/g for Hg(II), the KOH-AC showed the strongest affinity of 1.029±0.39 L/mg for Pb(II). This means that the KOH-AC could be very efficient for Pb(II) removal from very dilute AMD solutions (Oyewo et al., 2018).

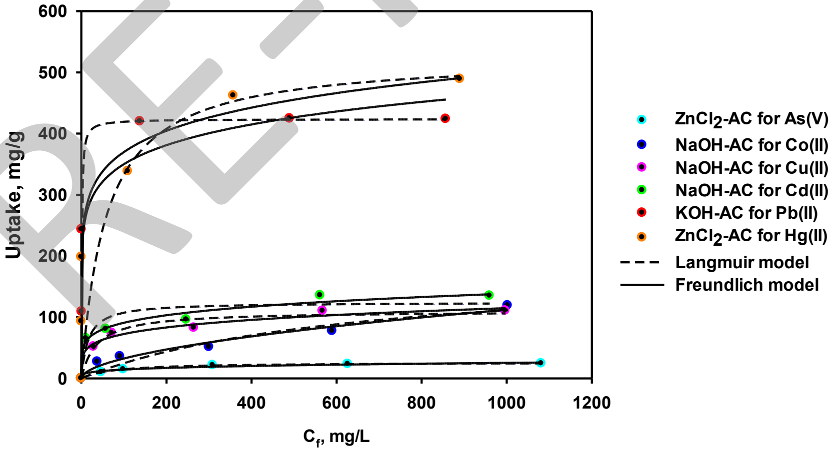


Figure 3

Isotherms of heavy metal adsorption based on the best suitable AC for each target metal. Experimental conditions: 10 mg AC in 20 mL of 0 – 1000 mg/L metal solutions at pH 6, 120 rpm, and 25±2 °C for 24 h

Table 2

Estimated parameters of the Langmuir isotherm model

Adsorbent-metal	**Langmuir Model**		
	q_m **(mg/g)**	***b*** **(L/mg)**	R^2
$ZnCl_2$-Hg	526.31±50.52	0.017±0.002	0.976
$ZnCl_2$-As	26.14±0.23	0.013±0.0005	0.999
KOH-Pb	426.62±20.68	1.029±0.39	0.993
NaOH-Cd	164.24±11.54	0.063±0.03	0.990
NaOH-Cu	129.95±6.31	0.028±0.008	0.986
NaOH-Co	141.75±26.01	0.002±0.0001	0.989

Table 3

Estimated parameters of the Freundlich isotherm model

Adsorbent-metal	**Freundlich Model**		
	k $(L/g)^{1/n}$	***n***	R^2
$ZnCl_2$-Hg	204.12±22.47	4.74±1.12	0.977
$ZnCl_2$-As	4.64±1.20	4.06±0.69	0.971
KOH-Pb	198.92±39.54	8.15±2.29	0.918
NaOH-Cd	39.16±6.42	5.47±0.83	0.979
NaOH-Cu	28.95±5.06	5.02±0.73	0.981
NaOH-Co	2.72±1.28	1.86±0.25	0.973

To provide information on the tendency of the adsorption process by the ACs, the dimensionless constant, R_L in Eq. (4) was derived from the Langmuir model and applied (Bediako et al., 2016; Yan et al., 2012).

$$R_L = \frac{1}{1 + bC_0} \tag{4}$$

where C_0 is the initial metal ion concentration (mg/L). The adsorption process can be described as either favorable ($0 < R_L < 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$), or irreversible ($R_L = 0$) (Bediako et al., 2016; Yan et al., 2012). Generally, smaller values of R_L ranging between 0 and 1 signify favorable adsorption with good binding affinity. The values obtained in this study were $0.1 < R_L < 0.9$ for the isotherm concentration range evaluated, i.e., 0 – 1000 mg/L, indicating that the adsorption process was favorable for the entire range of concentrations. The Freundlich model's n values further affirmed that the binding affinities were good and favorable, especially at higher initial concentrations (Yan et al., 2012).

3.4. Kinetics of competitive adsorption in multi-metal mixtures

Kinetics of competitive adsorptions in multi-metal solutions were conducted to estimate the rates of adsorption and identify any likely displacement mechanisms among the competing metal ions (Liu et al., 2008). A multi-metal mixture concentration of 100 mg/L excluding As was prepared. Here, the As (arsenic complex) was omitted due to its inducement of precipitation of other metal ions in the mixture. Similar to the isotherm results, the uptakes of Hg(II) and Pb(II) were significantly more compared to Cd(II), Cu(II) and Co(II). However, the adsorption rates were much faster in the case of the latter set, reaching equilibria within an average of ~1 h, whereas the equilibria of Hg(II) and Pb(II) were achieved between 5 – 10 h (Figure 4). This adsorption kinetics phenomenon could be understood to be probably due to the smaller atomic sizes of the latter set than the former set.

In a previous study using diethylenetriamine (DETA)-functionalized polymeric adsorbent, it was observed that initially adsorbed Pb(II) was displaced by subsequently adsorbed copper ions, likely because of the greater electronegativity of Cu(II) than Pb(II) (Liu et al., 2008). It was speculated that the displacement mechanism was possibly through the repulsion of Pb(II) and the adjacent attachment of Cu(II). However, this phenomenon was not observed in the present study. All the ACs were observed to show rising data points of uniform and increasing metal ion uptakes until equilibria were reached.

The kinetics data for each metal adsorption were fitted through the pseudo-first-order and pseudo-second-order models given by the following equations:

I) Pseudo-first-order:

$$q_t = q_1(1 - exp\,(-k_1\, t)) \tag{5}$$

II) Pseudo-second-order:

$$q_t = \frac{q_2^2 k_2 t}{1+ q_2 k_2 t} \tag{6}$$

where q_1 and q_2 are the uptakes at equilibrium given in mg/g; q_t is the adsorption capacity at time, t (mg/g); k_1 is the first-order equilibrium rate constant (min^{-1}), and k_2 is the second-order equilibrium rate constant (g/mg·min).

The two kinetic models closely fitted the data with relatively similar R^2 values in most cases; however, the pseudo-second-order provided fairly better fit. The ranges of R^2 values are summarized in Table 4. The similarities in the model fits depicted a combination of physisorption and chemisorption mechanisms by the ACs (Bediako et al., 2016). Furthermore, the estimated equilibrium uptakes were comparable, especially for the lowly adsorbed metals; however, in some cases, the predictions were underestimations of the actual uptake values, probably due to the rising trends of the data points.

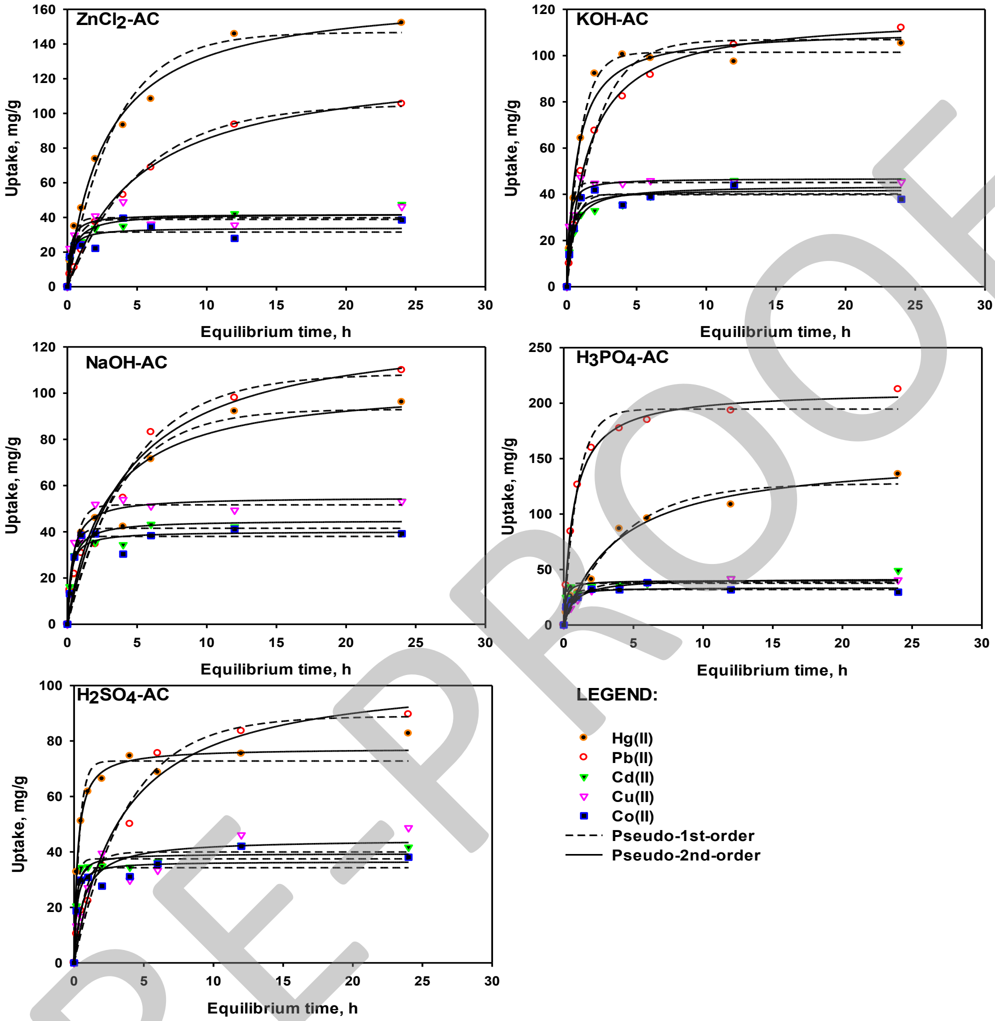


Figure 4

Kinetics of heavy metal adsorption in competitive metal mixtures. Experimental conditions: 0.1 mg ACs in 300 mL of 100 mg/L multi-metal solutions at 120 rpm and 25±2 °C for 24 h

Table 4

Ranges of R^2 values for fitted kinetics data from competitive adsorption

Metal	R^2 pseudo-first-order	R^2 pseudo-second-order
Hg(II)	0.895 – 0.996	0.925 – 0.998
Pb(II)	0.964 – 0.995	0.969 – 0.998
Cd(II)	0.833 – 0.953	0.922 – 0.968
Cu(II)	0.783 – 0.975	0.846 – 0.976
Co(II)	0.763 – 0.962	0.829 – 0.965

4. CONCLUSION

The adsorptive treatment of heavy metal ions contained in acid mine drainage (AMD) using fruit peel-derived ACs was studied. $ZnCl_2$-AC, KOH-AC, NaOH-AC, H_3PO_4-AC, and H_2SO_4-AC were effective for the removal of Hg(II), As(IV), Pb(II), Cd(II), Cu(II) and Co(II). The order of best adsorption capacity was $ZnCl_2$-AC for Hg(II) > KOH-AC for Pb(II) > NaOH-AC for Cd(II) > NaOH-AC for Co(II) > NaOH-AC for Cu(II) > $ZnCl_2$-AC for As(V), according to the isotherm studies. The kinetics of Cd(II), Cu(II), and Co(II) adsorption were faster than Hg(II) and Pb(II), likely because of the larger atomic sizes of the latter pair. The pseudo-second-order model provided a reasonably better fit to the kinetic data than the pseudo-first-order model. This study could be helpful for promoting sustainable mining practices through the adoption of effective AMD treatment strategies.

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