

Adsorption of copper, nickel and lead ions from synthetic semiconductor industrial wastewater by palm shell activated carbon

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ABSTRACT: Granular activated carbon produced from palm kernel shell was used as adsorbent to remove copper, nickel and lead ions from a synthesized industrial wastewater. Laboratory experimental investigation was carried out to identify the effect of pH and contact time on adsorption of lead, copper and nickel from the mixed metals solution. Equilibrium adsorption experiments at ambient room temperature were carried out and fitted to Langmuir and Freundlich models. Results showed that pH 5 was the most suitable, while the maximum adsorbent capacity was at a dosage of 1 g/L, recording a sorption capacity of 1.337 mg/g for lead, 1.581 mg/g for copper and 0.130 mg/g for nickel. The percentage metal removal approached equilibrium within 30 min for lead, 75 min for copper and nickel, with lead recording 100 %, copper 97 % and nickel 55 % removal, having a trend of $Pb^{2+} > Cu^{2+} > Ni^{2+}$. Langmuir model had higher R^2 values of 0.977, 0.817 and 0.978 for copper, nickel and lead respectively, which fitted the equilibrium adsorption process more than Freundlich model for the three metals.

Keywords: Granular activated carbon; Heavy metals; Isotherm; Sorption; Wastewater

INTRODUCTION

High concentration of heavy metals in the environment can be detrimental to a variety of living species. Excessive ingestion of these metals by humans can cause accumulative poisoning, cancer, nervous system damage and ultimately death (Corpacioglu and Huang, 1987; Issabayeva *et al.*, 2007). In Malaysia, industries dealing in electroplating, electronics, batteries and metal treatment/fabrication are the major sources of heavy metals contamination. Many of these industries are located in the western coast of the peninsular Malaysia, which includes Klang Valley, Malacca, Johor Bahru and Penang areas (DOE, 1979). Increased concern by environmentalists and governments on the effects of heavy metals and an attempt to protect public health has resulted in increased research in the development of advance technologies to remove heavy metals from waters and wastewaters (Bong *et al.*, 2004; Karbassi *et al.*, 2007; Shetty and Rajkumar, 2009; Resmi *et al.*, 2010). Such treatment efforts involved application of unit operations or unit processes such as chemical precipitation, coagulation, adsorption, ion exchange

and membrane filtration (Georg Steinhauser, 2008). Furthermore, among aforementioned treatment technologies, adsorption had been reported as an efficient and economic option (Skinner and Bassin, 1988; Mahvi, 2008; Malakootian *et al.*, 2009). A number of adsorbent materials have been studied for their ability to remove heavy metals and they have been sourced from natural materials and biological wastes of industrial processes (Igbinsosa and Okoh, 2009). These materials including: activated carbon (Uzun and Guzel, 2000; Goel *et al.*, 2005; Issabayeva *et al.*, 2007; Mondal *et al.*, 2008), chitosan and carrageenan (Bong *et al.*, 2004), lignite (Allen *et al.*, 1997), kaolinite and ballclay (Chantawong *et al.*, 2003), diatomite (Ulmanu *et al.*, 2003), coconut fiber (Igwe *et al.*, 2007) and limestone (Aziz *et al.*, 2004). However, adsorption by activated carbon had been reported as a technically and economically viable technology for heavy metal removal (Huang and Morehart, 1991; Bong *et al.*, 2004).

In Malaysia, the palm oil industry generates huge amounts of palm shell; a large portion of it is either burned in open air or dumped in area adjacent to the mill, which creates environmental and disposal problems. Therefore, application of palm shell activated

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carbon as an adsorbent offers highly effective technological means in dealing with pollution of heavy metals and solving palm shell disposal problems, with minimum investment required (Najua *et al.*, 2008). Pervious works on metals removal had focused on single metal ions removal and little information is available on multimetal adsorption (Abdel-Ghani and Elchaghaby, 2007; Abdel-Ghani *et al.*, 2009) even though most industries produce wastewater containing more than one metal ion in their effluent. In this work, investigation on the effect of pH, contact time and dosage on metal adsorption from a matrix of 3 metals was carried out using palm shell based activated carbon. While isotherm studies were used to model the adsorption process.

MATERIALS AND METHODS

Industrial wastewater samples from a semiconductor company (located at Kuala Langat, Malaysia) were collected for a week (8-12, June 2009), preserved and tested for metals content following standard method (Standard Methods, 2000) procedures. The concentration of heavy metals, namely lead, nickel and copper in the industrial wastewater served as the bases for the adsorption study in the laboratory.

Adsorbate

Metals solutions (copper, lead and nickel) of 1000 mg/L in nitric acid were purchased from Fisher Scientific, UK. Aqueous solutions of metals were prepared in the laboratory based on metal concentration in the industrial effluent. Each metal solution was diluted with distilled water to obtain the desired initial concentration, to mimic the industrial wastewater.

Adsorbent

The palm shell based granular activated carbon (GAC) was listed in Table 1 used as an adsorbent for this research was bought from KD Technology Malaysia. The adsorbent was washed with distilled water to remove fines and dirt following the procedure of Goel *et al.* (2005). The wet material was then dried at 110 °C for 6 h before characterization was done and used in the adsorption studies at ambient room temperature of 27 °C (± 2).

Adsorbent characterization

Scanning electron microscope (SEM) (JSM-5600 model) machine of the Japanese manufacturer (JEOL Ltd.) was employed to check the surface morphology of

Table 1: Properties of GAC adsorbents

Parameters	GAC
Raw material	Palm Kernel Shell
Particle size (mm)	1.68 -2.38
Ash content	4% max
Density (g/cm ³)	0.864
Surface area (m ² /g) (BET)	513.3
Average pore size (Å)	48.5
Total pore volume (cc/g)	0.622
Elemental analysis (%)	
C	64.45
N	23.09
O	0.00
Others	32.09

the GAC. The same machine was also used for the energy dispersive x-ray (EDX) spectra analysis to know the elemental composition of the GAC. Adsorbent samples for Fourier transform infrared (FTIR) analysis were prepared by diluting the GAC to 5 % in KBr and cast in disks for analysis to obtain the spectrum from a PerkinElmer precisely of model 100, earlier followed by Zvinowanda *et al.*, (2009). Specific surface area of the GAC was measured by an American Coulter sorptometer machine of model SA-3100, using N₂ as adsorbent at the liquid nitrogen temperature of -196 °C.

Adsorption study

For each experiment, 50 mL of synthetic water sample containing mixed metals of 2.0 mg/L Cu, 1.5 mg/L Pb and 0.8 mg/L Ni, was prepared based on industrial wastewater metal concentration and added to a calculated amount of adsorbent in 100 mL shake flask. The pH adjustment of solution was done using 1.0 M HCL and 1.0 M NaOH. The adsorbent in solution was agitated in a mechanical shaker at a speed of 100 rpm at 27 °C (± 2). Blank solutions were treated similarly without the adsorbent and under control condition. The solution was filtered using a Whatman[®] 0.45µm filter paper. The results were analyzed for the residual concentration of metals in the filtrate by atomic adsorption spectrophotometer (PerkinElmer HGA900).

Equilibrium concentration of metals at different adsorbent dosage (1, 2, 3, 4 g/L), at ambient temperature was used for the isotherm study. The adsorption capacity of adsorbent was calculated using Eq. 1.

$$q_t = \frac{(C_0 - C_t)V}{m} \quad (1)$$

Where, q_t is the adsorption capacity of the adsorbent at time t (mg adsorbate/g adsorbent); C_0 is



the initial concentration of metal (mg/L); C_t is the residual concentration of metal after adsorption had taken place over a period of time t (mg/L); V is volume of metal solution in shake flask (L) and m is mass of adsorbent (g) (Metcalf and Eddy, 2003). The metal removal percentage (R %) was calculated using Eq. 2.

$$R(\%) = \frac{(C_0 - C_t) \cdot 100}{C_0} \quad (2)$$

Where (R %) is the ratio of difference in metal concentration before and after adsorption.

RESULTS AND DISCUSSION

Physical and chemical characterization of adsorbent

The SEM image of Fig. 1 reveals the porous structure of the GAC surface. The FTIR spectra of GAC are shown in Fig. 2, while a summary of the peaks and their assignment are in Table 2. As can be inferred from the FTIR analysis, the acidic functional groups present on GAC surface were: carboxyl, carbonyl, lactones and sulphur groups. These groups had been reported to enhance metals adsorption (Zhang *et al.*, 2009; Edwin, 2008; Goel *et al.*, 2005). Specifically sulphur groups were reported by Goel *et al.* (2005) as aiding lead ions adsorption.

Effect of pH on adsorption

The uptake and percentage removal of metals from the aqueous solution are strongly affected by the pH of the solution (Horsfall and Abia, 2003; Zvinowanda *et al.* 2009). Fig. 3 shows the effect of pH variation on adsorption of copper, nickel and lead ions on GAC surface. The uptake of Cu, Ni and Pb increased from 0.095 mg/g to 0.685 mg/g; 0.045 mg/g to 0.137 mg/g and

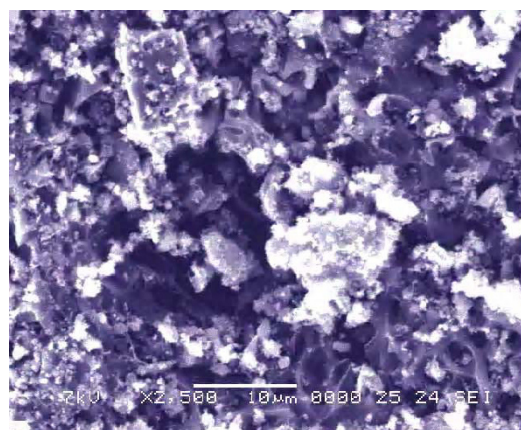


Fig. 1: SEM image of GAC surface

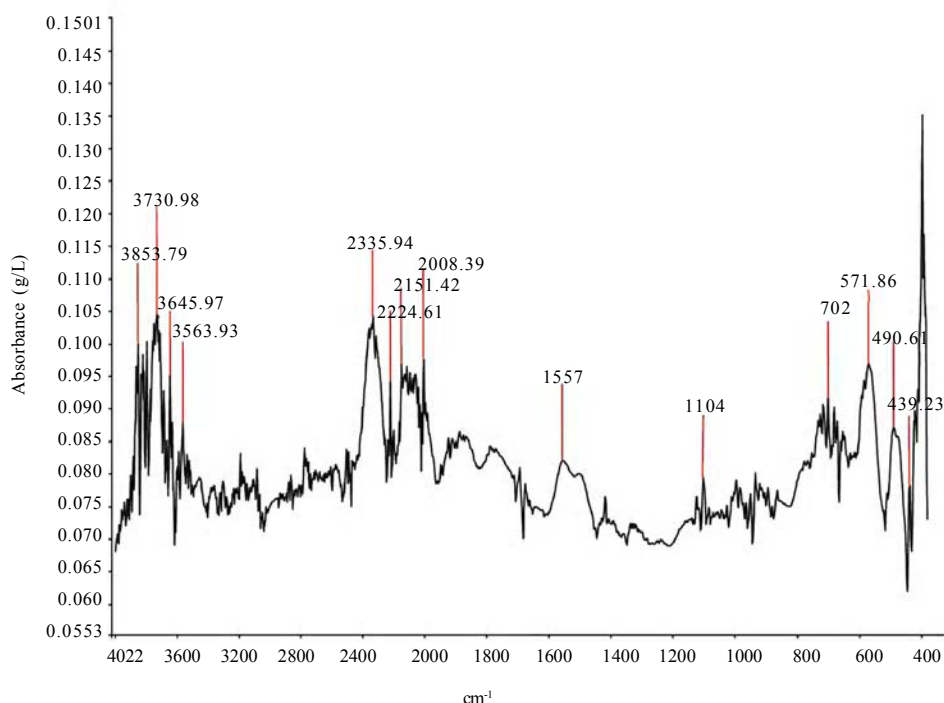


Fig. 2: FTIR spectrum of GAC



0.111 mg/g to 0.493 mg/g, respectively when the solution's pH was increased from 2 to 5. For pH values above 5, the adsorption capacity decreased with increment in pH. The mechanism of metals adsorption by the adsorbent with pH variation could be explained with the same concept as put forward by Corapcioglu and Huang (1987), Bansal and Goyal (2005), Najua *et al.* (2008) and that in the pH range of 2 to 3, copper, lead and nickel ions would be present predominately as M(II) ions. The minimum adsorption observed at low pH of 2 could be, on one hand due to the fact that the presence of higher concentration and higher mobility of H⁺ ions favoured H⁺ adsorption compared to M (II) ions and on the other hand due to the high solubility and ionization of metals salt in the acidic medium. It would be plausible to suggest that at lower pH value, the surface of the adsorbent is surrounded by hydronium ions (H⁺), thereby preventing metals ions from approaching the binding sites of the adsorbents. At higher H⁺ concentration, the adsorbents surface becomes more positively charged such that the attraction between adsorbents and metal cations is reduced. In contrast, as the pH increases, more negatively charged surface becomes available thus facilitating greater metals removal. It could be deduced that the sorption of metal cations increased with increasing pH as the metal ionic species become less stable in the solution. The maximum adsorption observed in the pH range of 4.5 to 6 might be due to partial hydrolysis of metal ions, resulting in the formation of M(OH)⁺ ions and M(OH)², which would be adsorbed to a greater extent on a less-polar carbon surface of the adsorbents compared to M²⁺ ions.

Furthermore, the low solubility of hydrolyzed metal species may be another reason for maximum adsorption in this pH range.

Effect of contact time

The relationship of percentage metals removal by adsorbent with contact time was plotted and presented in Fig. 4. The amount of the adsorbed metal ions increased as the time lapses, similar case was recorded by Gueu, *et al.*, (2007). The adsorbent showed a rapid metal reduction for the first 5 min of contact time, with further increase in time the sorption kinetics decreased progressively. The percentage metal removal approached equilibrium within 30 min for Pb and 75 min for Cu and Ni. The fast adsorption at the initial stage was probably due to the initial concentration gradient between the adsorbate in solution and the number of vacant sites available on the GAC surface at the beginning. The progressive increase in adsorption and consequently the attainment of equilibrium adsorption may be due to limited mass transfer of the adsorbate molecules from the bulk liquid to the external surface of GAC. It was noted that the sorption capacities

Table 2: Peaks from GAC spectrum and their possible assignment

GAC Band position/cm	Possible assignments
3564	O-H stretching (intermolecular diametric)
3200-2800	C-H stretching (alkenes)
2151	C=N stretching (alkynes)
1697	C=O Stretching (aldehydic)
1104	S-O Stretching (sulfates)
1125	C-H Amines
680-610	S-O Bends (sulfates)

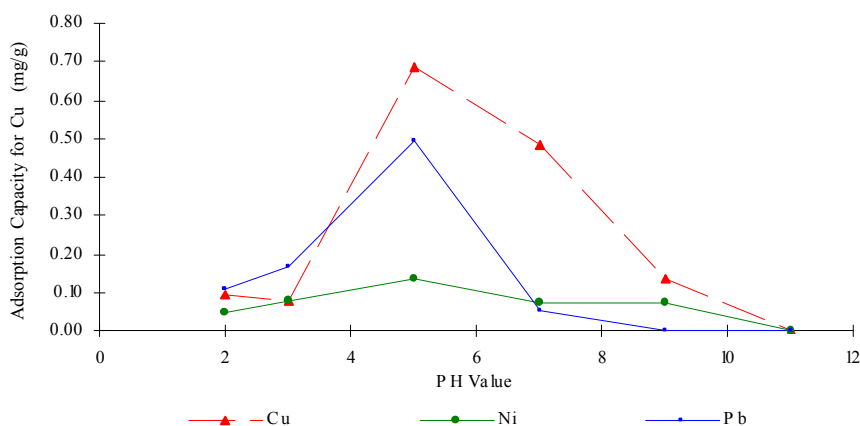


Fig. 3: Effect of pH on adsorption of metals onto GAC from a mixed metals solution



corresponding to equilibrium varied for each metal, with lead recording 100 %, copper 97 % and nickel 55 %, giving a trend of $Pb > Cu > Ni$ on the adsorbent. It was assumed that the acidic groups on the GAC had affinity for the three metals; the observed trend was probably due to the sulphate group on the adsorbent having higher affinity for lead ions, making lead reach equilibrium concentrations faster than copper and nickel ions. The higher initial concentration of copper also favored copper over nickel in the percent uptake achieved by 75 min. It is plausible to assume that the lower concentration of nickel probably did not favor nickel competition with lead and copper for the same available adsorption site on the GAC surface, which resulted in the comparable lower amount of nickel removal before equilibrium was attained.

Effect of adsorbent dosage

The effect of variation of GAC dosage on amount of metal adsorbed is shown in Fig. 5. A trend of increment in adsorption capacity with increment in adsorbent dosage was observed from 0.5 g/L to 1 g/L. Adsorbent recorded a maximum capacity of 1.581 mg/g for copper, 1.337 mg/g for lead and 0.130 mg/g for nickel at 1 g/L dosage. Further increment of adsorbent above 1 g/L resulted in a decline in adsorption capacity. The initial increment in adsorption capacity with increase in adsorbent dosage was expected, since number of adsorbent particles increases and thus more surface areas were available for metals attachment. Same trend was reported by Dakiky *et al.* (2002), Acharya *et al.*, (2009).

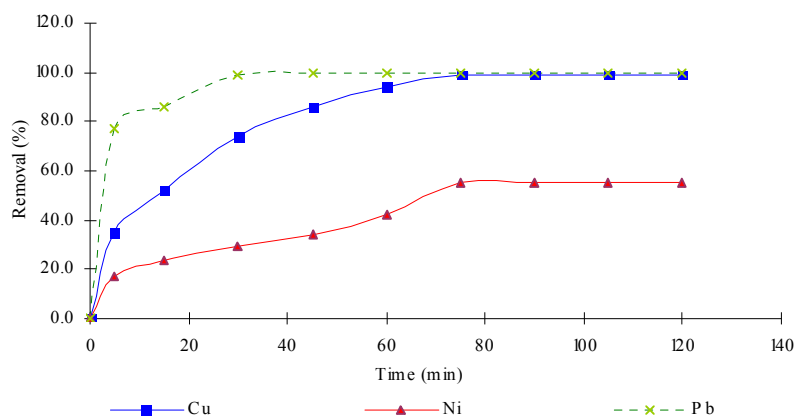


Fig. 4: Effect of contact time on adsorption of Cu (II), Ni (II) and Pb (II) (at ambient temperature =27 (±2)°C, adsorbent dose = 4 g/L, pH = 5).

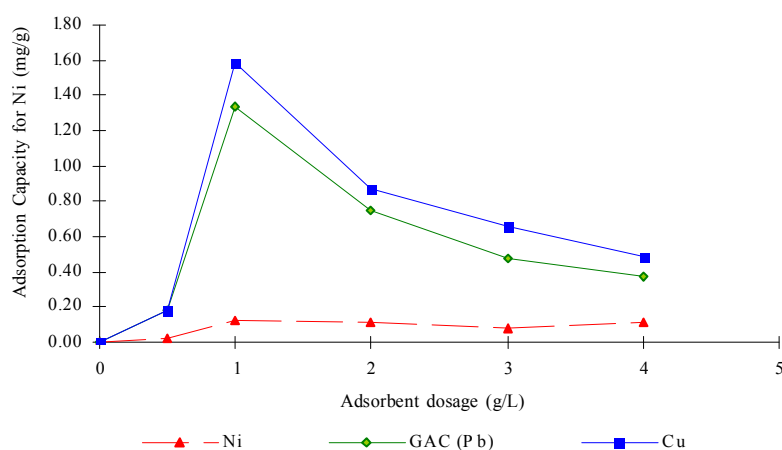


Fig. 5: Effect of adsorbent dosage (g) variation on metals adsorption (at pH5, agitation period of 75 min at 100 rpm)



It is plausible to suggest that with higher dosage of adsorbent there would be greater availability of exchangeable sites for metal ions as noted by [Babel and Kurniawan \(2004\)](#) and [Najua et al. \(2008\)](#). Further increment in adsorbent dosage beyond maximum adsorption capacity at 1 g/L resulted in a decline in capacity as shown in [Fig. 6](#). This reduction in capacity had been explained as due to the overlapping of the adsorption sites as a result of overcrowding of adsorbent particles beyond 1 g/L dose ([Garg et al., 2003](#); [Najua et al., 2008](#)). Moreover, the high adsorbent dosage could impose a screening effect on the dense outer layer of the cells, thereby shielding the binding sites from metals ([Pons and Fuste, 1993](#)).

Equilibrium adsorption isotherms

Several models had been used in literatures to describe the experimental data of adsorption isotherms. The Freundlich ([Freundlich and Hatfield, 1926](#)) and Langmuir ([Langmuir, 1918](#)) models are the most frequently employed models, these two models were used in the present work. Equilibrium data obtained were fitted to the Langmuir and Freundlich isotherms. The following expressions of straight line were found by means of mathematical transformation of isotherms equation.

Langmuir isotherm is shown as Eq.3:

$$c_s = \frac{1}{\frac{x}{m}} = \frac{1 + bc_e}{abc_e} = \frac{1}{a} + \frac{1}{abc_e} \tag{3}$$

Freundlich isotherm is shown as Eq. 4:

$$\log\left(\frac{x}{m}\right) = \log K_f + \frac{1}{n} \log C_e \tag{4}$$

Where, C_s is the amount of contaminant sorbed on the solid per unit of solid (mg sorbate/g sorbent), C_e is the concentration of contaminant remaining in

solution at equilibrium (g/L); x is the mass of contaminant adsorbed (g); m is the mass of solid sorbent (g) a and n are empirical coefficients; a (mg/g) in the Langmuir equation is indicative of the relative adsorption capacity of the adsorbent. The constant b is the saturation coefficient (m^3/g) and K_f is the Freundlich constant indicative of the relative adsorption capacity of the adsorbent (mg/g). The constant $1/n$ indicates the tendency of the adsorbate to be adsorbed ([Bishop, 2004](#); [Goel et al., 2005](#)). The estimated model parameters with correlation coefficient (R^2) for the two models are shown in [Table 3](#). It was observed that results fitted better in the Langmuir model in terms of R^2 value, recording 0.978 for lead, 0.977 for copper and 0.817 for nickel. The affinity of the metals for the adsorbent surface in terms of value “ b ” in the [Table 3](#) varied in the trend $Pb > Cu > Ni$, which is in agreement with the trend in [Fig. 4](#). It was assumed that this trend was due to the presence of sulphate acidic group present on the GAC surface. It was observed that the trend of adsorption capacity in terms of constant “ a ” was $Cu > Pb > Ni$. This was probably due to the initial concentration of metals following same trend and this trend is inline with trend in [Figs. 3 and 4](#). It could be inferred from this result that the trend of constant “ b ” was due to sulphate group present on the GAC, it also made Pb have higher affinity for the GAC surface, making it reach equilibrium concentration within 30 min of contact time, while Cu and Ni took longer time as was observed in [Fig. 4](#). However, the higher initial concentration of copper favored copper in total metal adsorbed on the GAC as shown in the trend of [Fig. 5](#), which also explains the trend of constant “ a ” in [Table 3](#).

Table 3: Isotherm parameters for metals adsorption at 27 (±2) °C and pH 5

Metals	Equation	Constants		Cs (Equation)	R ²
Copper	Langmuir	a =1.263	b=57.519	$c_s = \frac{x}{m} = \frac{(1.263)(57.519)}{1 + 57.519C_e}$	0.977
	Freundlich	Kf=0.228	n=3.777	$c_s = \frac{x}{m} = 0.228.c_e^{1/3.777}$	0.581
Nickel	Langmuir	a = 0.261	b=2.2951	$c_s = \frac{x}{m} = \frac{(0.261)(2.2951)}{1 - 2.2951C_e}$	0.817
	Freundlich	Kf=-0.607	n=1.29927	$c_s = \frac{x}{m} = -0.607.c_e^{1/1.29927}$	0.761
Lead	Langmuir	a = 0.841	b=80389.61	$c_s = \frac{x}{m} = \frac{(0841)(80389.61)}{1 + 80389.61C_e}$	0.978
	Freundlich	Kf= 0.112	n=8.4295	$c_s = \frac{x}{m} = 0.112.c_e^{1/8.4295}$	0.709



CONCLUSION

It was observed that copper, nickel and lead ions adsorption by GAC had highest adsorption capacity at pH 5, while the maximum adsorbent dosage was 1 g/L recording a sorption capacity of 1.337 mg/g for lead, 1.581 mg/g for copper and 0.130 for nickel. The metal removal approached equilibrium within 30 min for Pb, 75 min for Cu and Ni of contact time. Langmuir model having higher R² values of 0.977, 0.817 and 0.978 for copper, nickel and lead respectively fitted the equilibrium adsorption data more than the Freundlich model for the three metals. From the isotherm constants, it was observed that the trend of metals affinity for GAC surface was Pb > Cu > Ni, while the adsorbent metals affinity followed the trend Cu > Pb > Ni. This observed difference was assumed to be due to the sulphate functional groups present on GAC having higher affinity for Pb ions, while initial metal concentration favored Cu adsorption.

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