

Multi-component adsorption of copper, nickel and zinc from aqueous solutions onto activated carbon prepared from date stones

F. Bouhamed¹ · Z. Elouear¹ · J. Bouzid¹ · B. Ouddane²

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Abstract The removal of Cu^{2+} , Ni^{2+} , and Zn^{2+} ions from their multi-component aqueous mixture by sorption on activated carbon prepared from date stones was investigated. In the batch tests, experimental parameters were studied, including solution pH, contact time, initial metal ions concentration, and temperature. Adsorption efficiency of the heavy metals was pH-dependent and the maximum adsorption was found to occur at around 5.5 for Cu, Zn, and Ni. The maximum sorption capacities calculated by applying the Langmuir isotherm were 18.68 mg/g for Cu, 16.12 mg/g for Ni, and 12.19 mg/g for Zn. The competitive adsorption studies showed that the adsorption affinity order of the three heavy metals was $\text{Cu}^{2+} > \text{Ni}^{2+} > \text{Zn}^{2+}$. The test results using real wastewater indicated that the prepared activated carbon could be used as a cheap adsorbent for the removal of heavy metals in aqueous solutions.

Keywords Competitive adsorption · Activated carbon · Wastewater · Heavy metals

Responsible editor: Philippe Garrigues

✉ F. Bouhamed
bouhamed.farah@gmail.com

Z. Elouear
zouheir.elouaer@tunet.tn

J. Bouzid
bouzid.jalel@gmail.com

B. Ouddane
baghdad.ouddane@univ-lille1.fr

¹ Laboratoire Eau Energie et Environnement, Ecole Nationale d'Ingénieurs de Sfax, B.P.W 3038 Sfax, Tunisia

² Equipe Chimie Analytique et Marine, Université des Sciences et Technologies de Lille UMR CNRS—Lille1 8157 Geosystemes, Bât C8, 59655 Villeneuve d'Ascq Cedex, France

Introduction

The increasing levels of heavy metals in the environment represent a serious threat to human health, living resources, and ecological systems. Mobile and soluble heavy metal species are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders. Runoff water from industries, roads, and cities may contain high concentrations of heavy metals, and therefore, the removal of heavy metals from wastewaters is required prior to discharge into receiving waters (Moore and Ramamoorthy 1983; Kabata-Pendias and Pendias 1992; Álvarez-Ayuso et al. 2003; Inglezakis et al. 2004).

The adsorption is a separation process widely used for the removal of heavy metals from wastewater due to its versatility and easy operation (Corapcioglu and Huang 1987). Among the most commonly used adsorbents are activated carbon, biosorbents, natural zeolites, and clay minerals. The individual adsorption of heavy metals from water solution on these adsorbents has been extensively studied; however, industrial wastewater normally contains two or more metals. The presence of another metal in solution affects the adsorption capacity of the single metal (Benjamin and Leckie 1981). Therefore, it is very important to study the multicomponent adsorption of heavy metals and to find out about the selectivity or affinity of each metal for a given adsorbent and to know if there is competition between the metal cations for the same adsorption sites (Seo et al. 2008; Xiao and Thomas 2004).

Our recent work demonstrated a successful conversion of date stone residues to activated carbon (Bouhamed et al. 2012). The objective of this work was to determine sorption properties of the prepared activated carbon when contacted with a mixture of metal ions (Cu, Ni, and Zn), and thus, to extend our previous study made on single metal sorption on this material (Bouhamed et al. 2012; 2013a, b). A synthetic

mixture solution of copper, zinc, and nickel was arbitrarily selected as a modeled tertiary sorption system. Possible interactions between the metal ions and the surface of the adsorbent in the ternary component sorption were described.

Materials and methods

Adsorbent, chemicals, and equipment

In the present study, activated carbon was produced from Tunisian date stones. It was used as raw material to produce activated carbon via chemical activation using phosphoric acid (analytical grade) as a dehydrating agent. The activated carbon was prepared using a method described in a previous work (Bouhamed et al. 2012). The activated carbon has particle sizes ranging between 100 and 160 μm . The surface area (SBET), the total pore volume, and the average pore diameter of the prepared adsorbent, determined from the Brunauer–Emmett and Teller (BET) model, are $826 \text{ m}^2 \text{ g}^{-1}$, $0.49 \text{ cm}^3 \text{ g}^{-1}$, and 4.2 nm, respectively (Bouhamed et al. 2013a, b). The pH at point of zero charge (pHpzc) is 3.34, which indicates that the majority of the functional groups on the adsorbent are acidic.

Stock solutions for sorption were made by dissolving $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, and $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, in deionized water with different concentration (10, 30, 50, 70, and 100 mg/L). All chemicals were of analytical grade and used as received. Solution pH was adjusted using dilute nitric acid or sodium hydroxide.

The concentration of metals in the aqueous solution or in the aqueous phase after sorption was determined by the flame atomic absorption spectroscopy (HITACHI Z-6100). Analytical errors were estimated to be of the order of 3 %. All the experiments were duplicated to assure the veracity of the experimental results. Wave lengths used for the elements Cu, Ni, and Zn were 324.8, 228.8, and 213.9 nm, respectively.

Batch adsorption experiments

Adsorption experiments as a function of contact time, pH, concentration, and temperature were conducted. A known weight of activated carbon (0.5 g) was added to 200 mL of composite solutions containing equimolar concentrations of each metal ions (Cu(II), Ni(II), and Zn(II)) in the range of 10–100 mg/L. Before the addition, the pH of the metals solution was adjusted with diluted nitric acid or sodium hydroxide. The suspension was shaken over the desired time period, centrifuged, liquid supernatant was filtered through 0.2 μm membrane and concentration of metals in the filtrate was determined by AAS.

Experiments on sorption kinetics were carried out with the solution of initial concentration of 100 mg/L and contact time

of 5 to 480 min. The same initial concentration was used to study the effect of pH. To evaluate the effect of temperature, the sorption studies were also carried out at 10 and 40 $^\circ\text{C}$ by shaking in tempered water bath. All experiments were performed in triplicates; the standard deviation did not exceed 5 %.

Results and discussion

Sorption kinetics

At first, sorption kinetics was studied to determine the time necessary to attain equilibrium. Results show a rapid increase in Ni, Cu, and Zn adsorption that occurred within 30 min which may be explained by the presence of the number of available active sites in the activated carbon surface. There was no significant change in equilibrium concentration after 2 h which proves the saturation of the active sites (Fig. 1). Based on these results, a contact time of 2 h was selected in subsequent studies. The result obtained here is compatible with literature data and our previous experience with the activated carbon (Bouhamed et al. 2012, 2013a, b; Baccar et al. 2009; Vernersson et al. 2002; Delaila et al. 2008).

Effect of pH

The pH of the aqueous solution is an important controlling parameter in the adsorption process. The binding of metal ions by surface functional groups is strongly pH-dependent (Lee and Davis 2001). The effect of pH on the sorption of copper, nickel, and zinc is shown in (Fig. 2). As can be seen from the figure, sorbed amounts of all metal ions are low at low pH values due to the high concentration of hydrogen ions that compete with metal ions, which prevented the metal ions from approaching the binding sites on the adsorbent. The increase in metal removal as pH increases can be explained on the basis of a decrease in competition between hydronium ions and

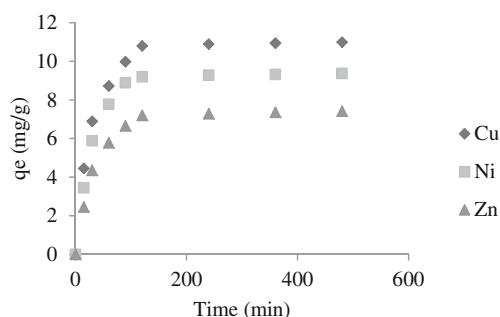


Fig. 1 Kinetic study for Cu, Ni, and Zn adsorption on the prepared activated carbon. Conditions: amount of activated carbon, 0.5 g; volume of solution, 200 mL; initial concentration, 100 mg/L; sorption time, 8 h; and temperature, 20 $^\circ\text{C}$

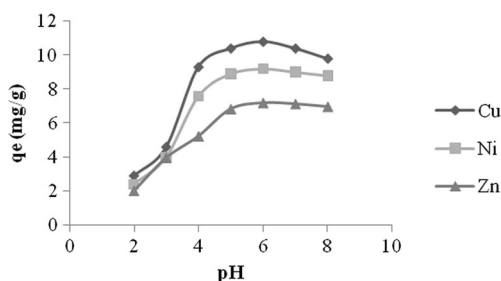


Fig. 2 Effect of solution pH on adsorption of Cu, Ni, and Zn on the prepared activated carbon. Conditions: amount of activated carbon, 0.5 g; volume of solution, 200 mL; initial concentration, 100 mg/L; sorption time, 2 h; and temperature, 20 °C

metal species for the surface sites and also by the decrease in positive surface charge on the adsorbent, which results in a lower electrostatic repulsion between the surface and the metal ions, and hence, the uptake of metal ions increases. The effect of pH can also be explained in terms of pH_{zpc} of the adsorbent. The pH_{zpc} of the adsorbent was 3.34. Below pH of 3.34, the surface charge of the activated carbon was also positive, and hence, the uptake of metals was low. When the solution pH was increased above 3.34, the surface of the activated carbon was negatively charged and thus the adsorption of metal ions increases. Optimal solution pH for sorption experiments was selected as 5.5 taking into account also hydrolysis and precipitation of metal cations. Similar conclusions have been reported also by other authors (Ho and McKay 1999; Zhu et al. 2012).

Sorption isotherms

The sorption isotherms were determined with solutions of initial concentration of each metal within the range 10–100 mg/L (at the pH of 5.5). The adsorption equilibrium isotherms obtained for Cu(II), Ni(II), and Zn(II) ions by activated carbon in ternary system are shown in (Fig. 3). Generally, the affinity of metal ions to activated carbon, as determined on the mass basis and from the positions of sorption isotherms in Fig. 3, decreases in the order: Cu>Ni>Zn, which was consistent with the order in single systems (Bouhamed et al. 2012, 2013a, b).

To describe experimental results, the original Langmuir and Freundlich isotherm equations were applied to determine the effect of presence of other metal ions on the isotherm constants. Other studies also used the original Langmuir and Freundlich models to fit the experimental data in binary and ternary systems (Futalan et al. 2011; Swayampakula et al. 2009). The Langmuir equation:

$$q_e = \frac{Q_0 b C_e}{1 + b C_e} \tag{1}$$

(q_e is the sorption capacity at equilibrium, Q_0 is the maximal sorption capacity, and b is the fitting parameter).

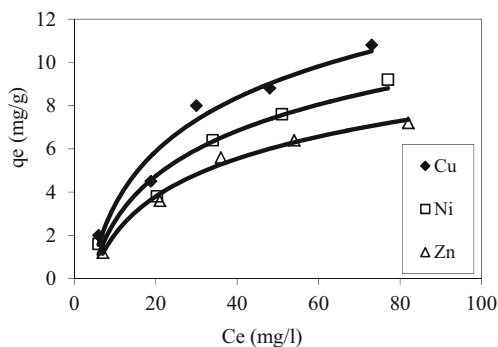


Fig. 3 Sorption isotherms for ternary metal system adsorption onto the prepared activated carbon. Conditions: amount of activated carbon, 0.5 g; volume of solution, 200 mL; sorption time, 2 h; pH 5.5; and temperature, 20 °C

The Freundlich isotherm:

$$q_e = K_f C_e^{1/n} \tag{2}$$

(c is the equilibrium concentration, and k and $1/n$ are the fitting parameters).

The competitive adsorptions of Cu(II), Ni(II), and Zn(II) on the adsorbent followed the Langmuir isotherm model very well with the values of R^2 (0.98–0.99) (Table 1), and they also fit the Freundlich isotherm model with the values of R^2 (0.95–0.97), but not better than the Langmuir isotherm model in general (Fig. 4a and b).

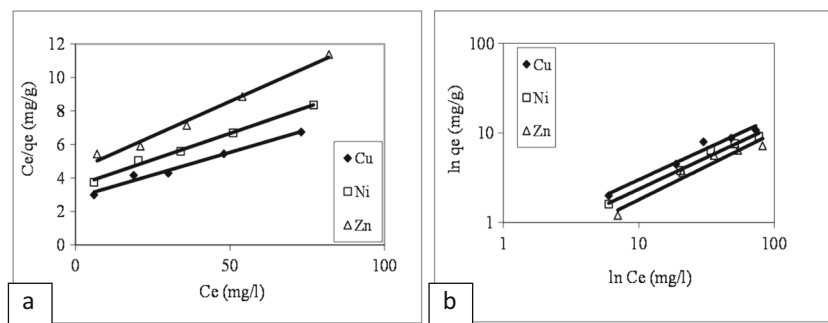
Maximum sorption capacities determined from the Langmuir model decreased in the order Cu>Ni>Zn both on the mass basis and on the molar basis. The maximum adsorption capacity in molar units was found for Cu, Ni, and Zn to be 0.29, 0.27, and 0.18 mmol/g, in the ternary mixture, respectively; while in the single metal system, they were 0.49, 0.27, and 0.33 mmol/g, respectively (Bouhamed et al. 2012; 2013a, b). The maximum sorption capacities for individual ions are smaller when sorbed from multi-metal solution which is clear indicator for the competition effects.

The mutual interference effects of metal ions on adsorption were probed using q_e'/q_e ratios, the q_e and q_e' , indicate the adsorption capacity at equilibrium in single and ternary systems. The value of q_e'/q_e will imply whether the effect of

Table 1 Adsorption isotherms model constants for ternary metal system adsorption on the prepared activated carbon

	The Langmuir isotherm constants			The Freundlich isotherm constants		
	Q_0 (mg/g)	b (l/mg)	R^2	K_f	$1/n$	R^2
Copper	18.68	0.018	0.99	0.6	0.69	0.96
Nickel	16.12	0.016	0.98	0.46	0.7	0.98
Zinc	12.19	0.018	0.98	0.32	0.74	0.95

Fig. 4 Langmuir and Freundlich adsorption of Cu(II), Ni(II), and Zn(II) onto the prepared activated carbon. Conditions: amount of activated carbon, 0.5 g; volume of solution, 200 mL; sorption time, 2 h; pH 5.5; and temperature, 20 °C



mixing metals in a solution is synergistic ($q_e'/q_e > 1$), no net interaction ($q_e'/q_e = 1$), or antagonistic ($q_e'/q_e < 1$; Mahamadi and Nharingo 2010). The q_e'/q_e values of Cu(II), Ni(II), and Zn(II) in ternary system were 0.59, 0.66, and 0.57, respectively. All the values were less than 1, implying that the adsorption of metal ions was suppressed by the presence of other metals in the solution. An antagonistic effect was exerted by the metal ions on others adsorption capacity. Prasad et al. (2008) studied the multi-component sorption of Pb(II), Cu(II), and Zn(II) onto low-cost mineral adsorbent using batch-type sorption experiments. The adsorption capacities of Pb(II), Cu(II), and Zn(II) followed the same order both in single system and ternary system. In ternary system, Cu(II) and Zn(II) were well fit both by the Langmuir and the Freundlich models, while Pb(II) correlated better with the Langmuir model. Liu et al. (2008) studied the competitive adsorption of heavy metals on peat. The results showed that the adsorption isotherm fit the Langmuir model very well. The adsorption capacities followed the order of Cu(II) > Ni(II) > Cd(II) in single-component systems and the competitive adsorption capacities fell in the decreasing order Cu(II) > Ni(II) > Cd(II) in multi-component systems. Doskočil and Pekař (2012) reported the removal of Pb^{2+} , Cu^{2+} , Cd^{2+} , and Zn^{2+} ions from their multi-component aqueous mixture by sorption on natural lignite. In multi-component system, the adsorption capacities was in the order of $Pb > Cu > Zn > Cd$ while in the single systems the order was $Pb > Cd > Cu > Zn$.

When multicomponent metal ions are present together, the sum adsorption capacity of total metal ions may increase, decrease, or not change (Hameed et al. 2007). The data in Table 1 indicated that total adsorption of Cu, Ni, and Zn on activated carbon was higher than that of in their single solute solutions in a previous work (Bouhamed et al. 2012; 2013a, b). Furthermore, in the Cu, Ni, and Zn multi-solute system, the total adsorption capacity was (46.99 mg/g) higher than Cu adsorption capacity (31.25 mg/g; Bouhamed et al. 2012), Ni adsorption capacity (24.4 mg/g), and Zn adsorption capacity (21.3 mg/g) in their respective single solute solutions in a previous work (Bouhamed et al. 2013b). These results suggest that the total adsorption capacity of heavy metals in multi-solute systems increases, but individual metal ion adsorption will decrease due to metal interaction and competition for

adsorption sites. This conclusion was in agreement with the work of Qin et al. (2006) and Hanzlik et al. (2004).

Effect of temperature and thermodynamic parameters

The effect of temperature from 10 to 40 °C on the adsorption capacity of activated carbon using 100 mg/L of Cu(II), Ni(II), and Zn(II) was investigated. The sorbed amounts slightly increased with increasing temperature for all metal ions. The increase in temperature from 20 to 40 °C increased the maximum sorption capacity, determined from the pseudo-Langmuir equation, from about 18.68 to 19.22 mg/g for Cu, from 16.12 to 16.94 mg/g for Ni and from 12.19 to 12.89 mg/g for Zn. From the trend, the increase of the uptake of metal ions by the activated carbon at higher temperature indicates that the adsorption is an endothermic process. The increase in the adsorption may be a result of increase in the mobility of the metal ions with increasing temperature. An increasing number of molecules may also acquire sufficient energy to undergo an interaction with active sites at the surface (Ucun et al. 2008) change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) for the sorption of metal ions on activated carbon was determined using the following equations (Sari et al. 2007; Chiou and Li 2003):

$$\Delta G^\circ = -RT \ln b \quad (3)$$

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \quad (4)$$

where ΔG° is the change in free energy (kJ/mol), ΔH° the change in enthalpy (kJ/mol), ΔS° the change in entropy (J/molK), T the absolute temperature K, R the gas constant (8.314×10^{-3}), and b is the equilibrium constant of sorption. From Eqs. (3) and (4), it can be rewritten as:

$$\ln b = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (5)$$

The enthalpy and entropy changes can be, respectively, determined from the slope and intercept of the plot $\ln b$ against $1/T$.

van't Hoff plots of Cu(II), Ni(II), and Zn(II) ions adsorption onto optimal activated carbon in multi-systems are depicted in Fig. 5. The calculated thermodynamic parameters

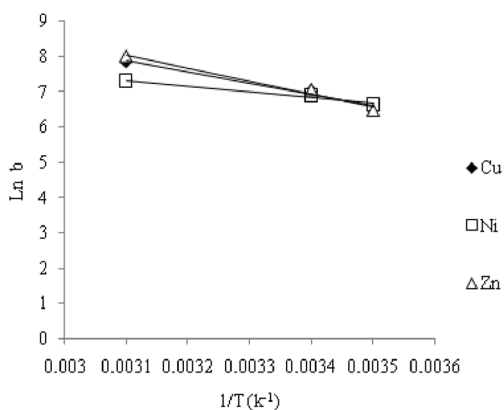


Fig. 5 van't Hoff plots of Cu(II), Ni(II), and Zn(II) adsorption on the prepared activated carbon

are summarized in Table 2. From 10 to 40 °C, the values of ΔG° for Cu(II), Ni(II), and Zn(II) are negative indicating that adsorption is spontaneous. This supports why the adsorption capacity of Cu(II) is higher than Zn(II) and Ni(II) because the more negative the ΔG° value, the more spontaneous and more favorable the adsorption. The enthalpy ΔH° values for Cu(II), Ni(II), and Zn(II) were found positive indicating that the adsorption process is endothermic; hence, increasing the temperature resulted to an increase in the metal adsorption capacity. The positive ΔS° values signify there is an increase in the randomness at the solid-solution interface of Cu(II), Ni(II), and Zn(II) onto activated carbon surface (Bayramoğlu and Arica 2008).

Treatment of metal industrial wastewater

Based on the promising results of heavy metal removal from aqueous solutions, tests were conducted to evaluate these results using real wastewater. The wastewater used was taken from a local metal plating factory. It had a pH of 6 and contained Zn of 8.23 mg/L, Ni of 1.89 mg/L, and Cu of 1.74 mg/L. Tests were conducted at 25 °C for different contact time (5 to 480 min) with activated carbon concentration of

Table 2 Thermodynamic parameters for the adsorption of Cu(II), Ni(II), and Zn(II) ions on activated carbon

Temperature (°C)		10	20	40
Cu(II)	ΔG° (Kcal/mol)	-3.94	-4.08	-4.36
	ΔH° (Kcal/Kmol)	6.44	6.44	6.44
	ΔS° (cal/(Kmol))	35.61	35.61	35.61
Ni(II)	ΔG° (Kcal/mol)	-3.86	-4	-4.27
	ΔH° (Kcal/kmol)	3.22	3.22	3.22
	ΔS° (cal/(Kmol))	24.51	24.51	24.51
Zn(II)	ΔG° (Kcal/mol)	-3.96	-4.01	-4.38
	ΔH° (Kcal/Kmol)	7.16	7.16	7.16
	ΔS° (cal/(Kmol))	38.11	38.11	38.11

2.5 g/L. At the end of the period, the solutions were separated by filtration and the residual metal concentration in the supernatant was determined by atomic absorption spectrophotometry.

The results of the tests using real wastewater (Fig. 6) showed that activated carbon was effective in the simultaneous removal of various heavy metals in metal industrial wastewater. It was found that maximum removal of Zn, Ni, and Cu was reached within 2 h. In addition, the residual Zn, Ni, and Cu concentrations in solution (1.2, 0.08, and 0.07 mg/L, respectively) at the equilibrium time was below the industrial effluent standard (NT. 106.002 (1989) set by Ministry of Health, Economy, Agriculture and Environmental in Tunisia (5, 2, and 1 mg/L, respectively).

Conclusions

Sorption experiments for copper, nickel, and zinc ions adsorption on activated carbon in ternary system were carried out in the batch mode. Concentration of metal ions in supernatant was close to equilibrium within 2 h and the optimal pH for the sorption was determined to be about 5.5. Adsorption isotherms clearly indicated competitive effects upon sorption. The order of affinity decrease for metal ions sorption on activated carbon calculated on the mass basis is as follows: Cu > Ni > Zn. The data could be fitted by both Freundlich and pseudo-Langmuir isotherm models, the latter giving somewhat better results. The maximum adsorption capacities calculated by the pseudo-Langmuir equation were 18.68, 16.12, and 12.19 mg/g for the Cu, Ni, and Zn ions, respectively. The increase in temperature resulted in an increase of sorption of copper, nickel, and zinc ions. The results of this study showed that the prepared activated carbon was suitable for the removal

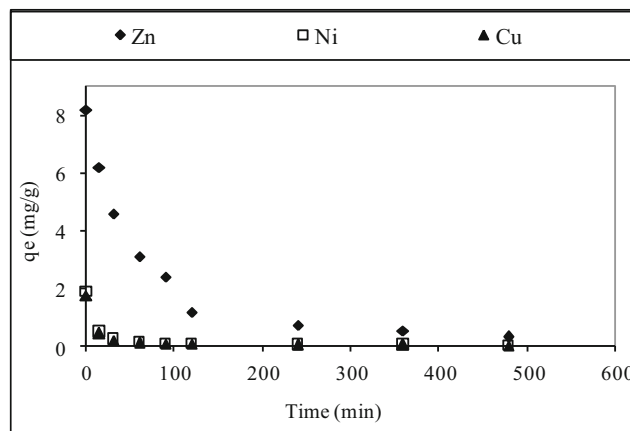


Fig. 6 Removal of various heavy metals in metal industrial wastewater by the prepared activated carbon. Conditions: amount of activated carbon, 0.5 g; volume of solution, 200 mL; sorption time, 8 h; pH 6; and temperature, 25 °C

of metal ions present in multicomponent system in contaminated wastewater.

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