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ORIGINAL PAPER

Removal of heavy metals from wastewater using infiltration-percolation process and adsorption on activated carbon

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Abstract
Heavy metal pollution has become one of the most serious environmental problems nowadays. The removal of heavy metals from wastewaters has attracted a considerable attention because of their adverse effects on public health and ecosystems. The main objective of this work was to investigate the efficiency of the coupling of infiltration-percolation process with adsorption on activated carbon in the removal of heavy metals contained in urban wastewater effluents. The adsorption of heavy metals on a commercial sample of activated carbon was studied in a static mode. Several laboratory experiments made it possible to distinguish the optimum quantity of powdered activated carbon necessary to remove a large range of heavy metals. Results showed that the equilibrium of the adsorption was reached very quickly for cadmium (Cd^{2+}), i.e., after 15 min of contact with the activated carbon. On the other hand, the equilibrium of zinc (Zn^{2+}), lead (Pb^{2+}) and copper (Cu^{2+}) was achieved after 45 min. The withdrawal rates were 70.77% for Zn^{2+}, 64.75% for Pb^{2+}, 67.07% for Cu^{2+} and 78.42% for Cd^{2+}. The adsorption isotherms determined for Zn^{2+}, Pb^{2+}, Cu^{2+} were of type I, while the shape of the Cd^{2+} curve showed a type II isotherm. These isotherms confirm the capacity of the powdered activated carbon to adsorb cadmium better than the other studied heavy metals.

Keywords Adsorption · Activated carbon · Coupling · Heavy metals · Infiltration-percolation

Introduction

Heavy metals are dangerous species which induce various environmental and health problems (Göde et al. 2017). Most industries unload wastewater and effluent containing toxic materials, mainly heavy metals, into aquatic systems without treatment, which is a major environmental problem. The excessive release of these pollutants presents a particular problem because they are not biodegradable (Boussahel 2001). These pollutants do not all have the same risks to the environment (Houas et al. 1999) and to health because of their different physicochemical properties.

Effluents are still treated at wastewater treatment plants (WWTPs), apart from exceptional cases of storm spills, and thus the future of the toxic pollutants contained in wastewater will very much depend on the purification efficiency of these stations (Varrault 2011). This author has shown that during the treatment, a small quantity of metallic trace elements is not eradicated and will, therefore, pass into the aquatic environment. Furthermore, the uppermost proportion of metals is trapped in the sewage sludge and can, therefore, be transferred either to unloading stations or to agriculture which receives this type of sludge. Heavy metals can reach concentrations that threaten the survival of certain populations and pose dangers. Indeed, heavy metals in water are absorbed by plants and animals. When the amount of these elements exceeds the norms, they can accumulate in the organisms of the food chain (Lacoue-Labarthe 2007).

There are several methods for heavy metal detection including inductively coupled plasma-mass spectrometry (Liao and Jiang 1999), spectrophotometry (Jankiewicz et al. 2000), flame-atomic absorption spectrometry (Bortoleto et al. 2004) or electrochemical methods that include ion-selective electrodes, polarography and other modified electrodes (Yola et al. 2012; Gupta et al. 2013a, b; Yola et al. 2014; Göde et al. 2017). High sensitivity and high speed of

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analysis are provided with electrochemical methods (Gupta et al. 2013a, b).

The withdrawal of inorganic compounds, such as heavy metals dissolved in water, requires the use of treatment processes such as adsorption, membrane separation, coagulation-flocculation, precipitation, ion exchange and filtration (Boulkrah 2008). However, these methods were deemed limited since they often involve high costs of exploitation and regeneration which are the current problems of treatment, mainly in poor countries like Tunisia. Adsorption is effective compared to other methods (Escher and Leusch 2011). Indeed, a good adsorption power is obtained with materials having a high specific surface area, such as activated carbon. The degree of adsorption of micropollutants depends on the type of active carbon (porosity, surface, surface functional groups, etc.), characteristic of micropollutants (molecular structure, size, hydrophobicity, etc.), contact time and dose of coal, etc. (Margot et al. 2011). Powdered activated carbon (PAC) is generally preferred because of its adsorption capacity and adsorption rate which are generally higher compared to granular activated carbon (Margot et al. 2011).

Activated carbon is used in various fields (Bouziane 2007), such as tertiary treatment of urban wastewater. However, the saturation of the surface of this material by the adsorption of a solute limits its duration of use following the accumulation of pollutants transported by wastewater such as suspended solids, organic matter and nitrogen pollution as well as biological pollution that compete with heavy metals over the pores of activated carbon. In this case, treatment with activated carbon should be considered as a complement to the biological purification of wastewater which allows improving the removal of toxic non-biodegradable compounds. It is therefore necessary that the treatment of secondary effluents is accompanied in the first place by a treatment phase capable of eliminating a large range of pollutants. Infiltration-percolation is an extensive treatment process aiming to remove organic pollution, ammonium and pathogens (Al 1999). This process has been applied as a tertiary treatment of secondary effluents in a number of Mediterranean countries (Bali et al. 2011). It involves the infiltration of primary or secondary wastewater into ponds unearthed in the soil or filled with sand (Nadav et al. 2017). Several authors (Oladoja and Ademoroti 2006; Akadar 2014) reported that the elimination of pollutants from wastewater depends on the characteristics of the filter media.

The infiltration-percolation process has been progressively used for the treatment of wastewater effluents due to its low-energy and maintenance requirements (Asano and Cotruvo 2004; Stevik et al. 2004). For this reason, the objective of the present work was to couple infiltration-percolation process with adsorption on PAC in order to improve the removal of heavy metals such as zinc (Zn^{2+}), lead (Pb^{2+}), copper ($Cu^{2+)}$ and cadmium (Cd^{2+}).

The phenomenon of adsorption is generally explained by curves giving the quantity of gas or adsorbed solute as a function of the concentration of the solutes or of the relative pressure of the gas. However, according to IUPAC (1985) and Dabrowski (2001), the most commonly used adsorption equilibrium is the adsorption isotherm. The vast majority of isotherms can be classified into six types according to their rhythms (Slasli 2002). However, it is possible to find several variants for each type of isothermal classification or for isotherms representing combinations of isotherms of the conventional type (Brunauer 1944). Each type of isotherm gives qualitative conclusions on the interactions between the adsorbates and the adsorbent (Jankowska et al. 1991).

Materials and methods

The experimental study lasted for a period of 6 months, from April to October 2016. The experiments were carried out in the laboratory of the Applied Hydro-Sciences of the Higher Institute of Sciences and Techniques of Waters (University of Gabès, Tunisia).

In order to determine the best wastewater treatment process for the removal of heavy metals, the wastewater has been treated successively by infiltration-percolation and adsorption on powdered activated carbon.

Experiments were carried out on polyvinyl chloride (PVC) column of 200 cm in height and 10 cm in diameter (Fig. 1). The bottom of the column was charged with a layer of coarse gravel surmounted by 1.5 cm of sand whose average size (d_{50}) was 0.26 mm and which had great uniformity (Cu = 1.93). The infiltration experiments were carried out using a secondary effluent from the wastewater treatment plant of the city of Gabès (southeast of Tunisia). The applied hydraulic load was 0.27 m/day. It was selected according to the works carried out by several authors at a laboratory scale column (Guediri 2000; Bali et al. 2016). The infiltration system was carried out according to an operation mode rotated through a 7-day cycle consisting of a 4-day feeding period and a 3-day drying period to avoid the clogging of the filter bed. This operation allows the adjustment of the biomass accumulated in the filter, which facilitates endogenous respiration.

At the outlet of the sand filter, the treated effluent samples were coupled with an amount of PAC selected in the following manner: One liter of water to be treated was introduced into four beakers filled with four quantities of the adsorbent (15, 25, 35 and 45 g) for the purpose of testing the most suitable quantity to eliminate a wide range of pollutants. Stirring was adjusted to 300 rpm, and the water samples were taken at 15-min intervals and then filtered on a filter paper and analyzed by specific electrode techniques to measure the concentration of each metal concentration relative to the concentration of the

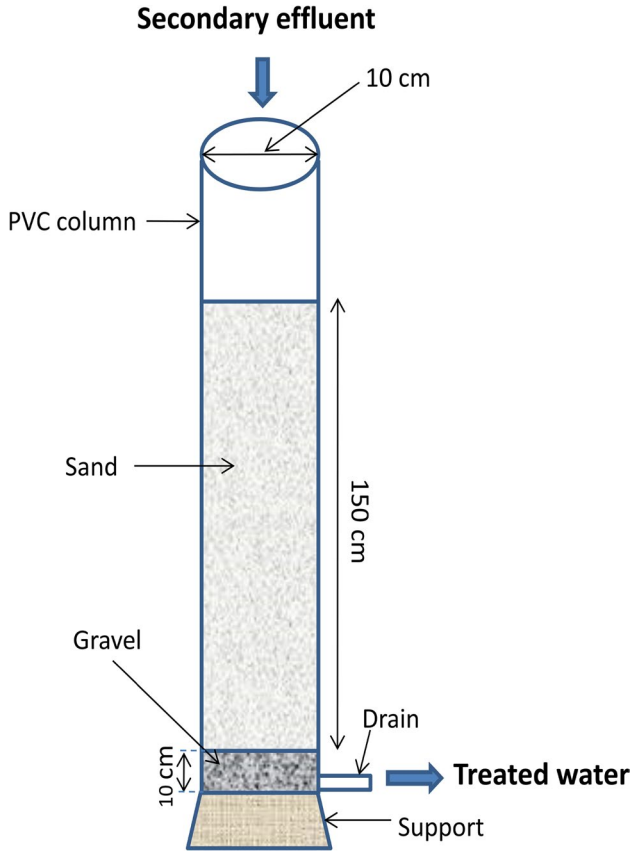

Fig. 1 Simplified diagram of the infiltration-percolation column

initial sample. The quantity of the PAC corresponding to the best reduction of heavy metals was then adapted in the process of coupling with the infiltration-percolation process.

To better interpret the phenomenon of adsorption of heavy metals by the coupling of these two treatment processes, an isothermal study was conducted by modeling the adsorption equilibrium of each metal. The adsorption capacity of the solid is given by the following equation (Boulkrah 2008):

$$Q = x/m = (C_i - C_e)v/m \quad (1)$$

where x is the mass of the adsorbate, m is the mass of the adsorbent, v is the volume of the applied solution, and C_i and C_e are the initial and the equilibrium concentrations of the solute.

This method is often applied to determine the adsorbed quantities on solid surfaces. The adsorption isotherms are given by curves in the form:

$$Q_e = f(C_e). \quad (2)$$

Table 1 Variation of the Zn^{2+} concentration as a function of time and PAC mass as well as the removal rate

m (g)	T (min)	C_i (ppm)	C_e (ppm)	R (%)
15	15	0.0283	0.0128	54.77
	30		0.0126	55.47
	45		0.0132	53.35
	60		0.0129	54.41
	75		0.0122	56.89
	90		0.0119	57.95
	105		0.0123	56.53
	120		0.0120	57.60
25	15	0.0283	0.0133	53.00
	30		0.0132	53.35
	45		0.0125	55.83
	60		0.0124	56.18
	75		0.0133	53.00
	90		0.0129	54.41
	105		0.0150	46.99
	120		0.0147	48.05
35	15	0.0283	0.0141	50.17
	30		0.0128	54.77
	45		0.0116	59.01
	60		0.0128	54.77
	75		0.0105	62.89
	90		0.0116	59.01
	105		0.0126	55.47
	120		0.0149	47.34
45	15	0.0283	0.0105	62.89
	30		0.0106	62.54
	45		0.0104	**63.25**
	60		0.0106	62.54
	75		0.0106	62.54
	90		0.0107	62.19
	105		0.0119	57.59
	120		0.0117	58.65

Bold value indicates the best removal rate

m mass of PAC, T time, C_i initial concentration, C_e equilibrium concentration, R removal rate

Results and discussion

Evaluation of the adsorption of heavy metals on PAC

In order to choose the optimum quantity of PAC to be adapted during the coupling with the infiltration-percolation process, analyses were carried out on different masses of PAC to determine the most adequate quantity for the abatement of heavy metals. Tables 1, 2, 3 and 4 show the results of analyses of the four heavy metals tested.

Table 2 Variation of the Pb^{2+} conc entration as a function of time and PAC mass as well as the removal rate

m (g)	*T* (min)	C_i (ppm)	C_e (ppm)	*R* (%)
15	15	0.3931	0.2626	33.19
	30		0.2579	34.39
	45		0.2706	31.16
	60		0.2651	32.56
	75		0.2512	36.09
	90		0.2458	37.47
	105		0.2524	35.79
	120		0.2478	36.96
25	15	0.3931	0.2729	30.57
	30		0.2708	31.11
	45		0.1813	53.87
	60		0.2546	35.23
	75		0.2736	30.39
	90		0.2644	32.73
	105		0.3078	21.69
	120		0.2999	23.70
35	15	0.3931	0.2882	26.68
	30		0.2641	32.81
	45		0.1699	56.77
	60		0.2632	33.04
	75		0.2154	45.20
	90		0.2391	39.17
	105		0.2583	34.29
	120		0.3037	22.74
45	15	0.3931	0.2135	45.68
	30		0.2143	45.48
	45		0.1580	**59.80**
	60		0.2189	44.31
	75		0.2199	43.78
	90		0.2210	44.06
	105		0.2210	43.78
	120		0.2409	38.71

Bold value indicates the best removal rate

m mass of PAC, *T* time, C_i initial concentration, C_e equilibrium concentration, *R* removal rate

Table 3 Variation of the Cu^{2+} concentration as a function of time and PAC mass as well as the removal rate

m (g)	*T* (min)	C_i (ppm)	C_e (ppm)	*R* (%)
15	15	0.3074	0.1862	39.42
	30		0.1829	40.50
	45		0.1920	37.54
	60		0.1880	38.84
	75		0.1780	42.09
	90		0.1742	43.33
	105		0.1790	41.76
	120		0.1756	42.87
25	15	0.3074	0.1936	37.02
	30		0.1922	37.47
	45		0.1813	41.02
	60		0.1804	41.31
	75		0.1941	36.85
	90		0.1876	38.97
	105		0.2187	28.85
	120		0.2130	30.70
35	15	0.3074	0.2047	33.40
	30		0.1874	39.04
	45		0.1699	44.73
	60		0.1866	39.29
	75		0.1524	50.42
	90		0.1694	44.89
	105		0.1831	40.43
	120		0.2158	29.79
45	15	0.3074	0.1510	50.87
	30		0.1516	50.68
	45		0.1201	**60.93**
	60		0.1549	49.60
	75		0.1557	49.35
	90		0.1565	49.08
	105		0.1610	47.62
	120		0.1706	44.50

Bold value indicates the best removal rate

m mass of PAC, *T* time, C_i initial concentration, C_e equilibrium concentration, *R* removal rate

Figure 2 illustrates the variation of these heavy metals as a function of the time and the mass of the added PAC. In fact, the results obtained show a significant difference in the concentrations of the heavy metals contained in the wastewater and the water purified by the different masses of PAC (Tables 1, 2, 3, 4). The concentration of adsorbate affects the adsorption capacity. The quantities of the adsorbed heavy metals increased with the PAC mass dissolved to stabilize at a mass of 45 g. This yielded better elimination rates, i.e., 63.25, 59.80, 60.93 and 73.56%, respectively, for Zn^{2+}, Pb^{2+}, Cu^{2+} and Cd^{2+}.

The adsorbed quantities increased as a function of the mass of PAC regardless of time. For the different stirring times (15, 30, 45, 60, 75, 90, 105 and 120 min), the equilibrium concentrations of Zn^{2+} were lower by adding 45 g/l of PAC (Fig. 2a), resulting in higher removal rates (62.89, 62.54, 63.25, 62.54, 62.54, 62.19, 57.59 and 58.65%, respectively). As can be seen from this figure, the variation of these values leads to a fluctuation of the equilibrium concentrations. This can be explained by a clogging which results from the interactions between the pollution carried by the secondary effluent and the heavy

Table 4 Variation of the Cd^{2+} concentration as a function of time and PAC mass as well as the removal rate

m (g)	*T* (min)	C_i (ppm)	C_e (ppm)	*R* (%)
15	15	0.0087	0.0034	60.91
	30		0.0033	62.06
	45		0.0036	58.62
	60		0.0034	60.91
	75		0.0032	63.21
	90		0.0030	65.51
	105		0.0032	63.21
	120		0.0031	64.36
25	15	0.0087	0.0037	57.47
	30		0.0036	58.62
	45		0.0032	63.21
	60		0.0032	63.21
	75		0.0037	57.47
	90		0.0034	60.91
	105		0.0044	49.52
	120		0.0043	50.57
35	15	0.0087	0.0040	60.91
	30		0.0034	39.04
	45		0.0029	66.66
	60		0.0034	60.91
	75		0.0024	72.41
	90		0.0029	44.89
	105		0.0033	62.06
	120		0.0044	49.42
45	15	0.0087	0.0023	**73.56**
	30		0.0024	72.41
	45		0.0024	72.41
	60		0.0024	72.41
	75		0.0025	71.26
	90		0.0025	66.66
	105		0.0026	70.11
	120		0.0030	65.51

Bold value indicates the best removal rate

m mass of PAC, *T* time, C_i initial concentration, C_e equilibrium concentration, *R* removal rate

metals at the adsorption sites of the PAC. After 45 min and with 45 g of PAC, a maximum elimination of 63.25% was attained. The adsorption of zinc can be achieved according to two mechanisms: either by cation exchange or by chemisorption, in which there is a formation of bonds between the specific surface area of the atoms and the molecules of the adsorbate (Babié et al. 2002).

The adsorption capacity of lead also depends on the mass of the PAC capable of eliminating large amounts of this micropollutant (Fig. 2b). In addition, the best elimination rates obtained at 45 g/l of PAC were, respectively, 45.68, 45.48, 59.80, 44.31, 43.78, 44.06, 43.78 and 38.71% (Table 3). Lead was trapped by simple adsorption on the surface of activated carbon or by precipitation. Less efficient results were obtained by Boulkrah 2008 when removing lead contained in an artificial solution synthesized using a different PAC.

Similarly, the highest elimination results for copper were obtained by adding a mass of 45 g of PAC along the chosen contact times (50.87, 50.68, 60.93, 49.60, 49.35, 49.08; 47.62 and 44.50%, respectively) (Fig. 2c). In general, the adsorption capacity of the adsorbent decreases with the solid/liquid ratio.

For the three heavy metals (Zn^{2+}, Pb^{2+} and Cu^{2+}), the equilibrium was attained after 45 min with a mass of 45 g/l of PAC. These are the two optimal parameters (time, mass) to adapt during the coupling of infiltration-percolation and adsorption on PAC processes.

According to Creanga (2007), the phenomenon of adsorption is carried out in several stages of diffusion of the molecules; it can be extended over very long times according to the nature of the adsorbent and the diameter of the molecules of the adsorbate. It can also reach its equilibrium very quickly (few seconds to few minutes). This is the case of the adsorption of Cd^{2+} contained in the secondary effluent. Indeed, unlike the other heavy metals tested, the elimination of cadmium was achieved after the first 15 min with an adsorbent mass of 45 g. This mass results in a maximum removal of 73.56%. The adsorption of this pollutant remains very easy because its concentration is low in the sample compared to other heavy metals. It had an initial concentration of 0.0087 ppm and decreased to 0.0023 ppm (Fig. 2d) after analysis on PAC. It is therefore a physical adsorption. According to Salsli (2002), physisorption is of particular interest because it makes it possible to measure the specific surface area of the solid adsorbent and the average pore size according to some criteria, such as rapidity of the adsorption process in the case of cadmium.

In general, the search for the adsorption kinetics is a prerequisite for the determination of the optimum mass of the PAC which makes it possible to eliminate a wide range of heavy metals as a function of time. The results showed that cadmium has more adsorption affinity on PAC than the other studied heavy metals which facilitates its attachment to the specific surface area of this adsorbent ($Cd^{2+} > Zn^{2+} > Cu^{2+} > Pb^{2+}$) (Fig. 3).

Study of the efficiency of the coupling of infiltration-percolation and adsorption on PAC in the treatment of urban wastewater

Table 5 shows the static data of the analysis results for the heavy metal contents of the secondary effluent, the percolate from the infiltration-percolation column and the water

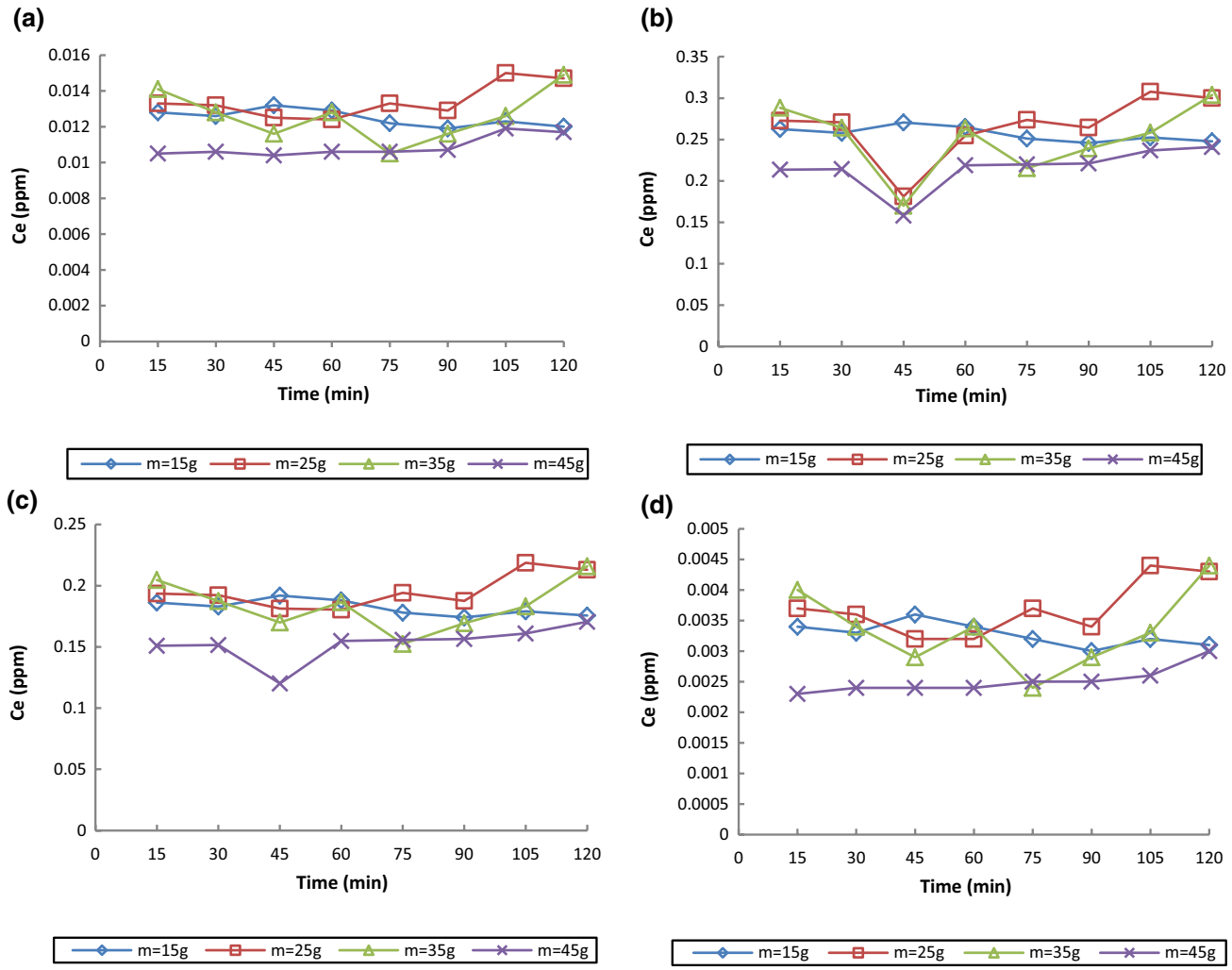

Fig. 2 Variation of the concentrations of Zn^{2+} (**a**), Pb^{2+} (**b**), Cu^{2+} (**c**) and Cd^{2+} (**d**) as a function of time and powdered activated carbon mass

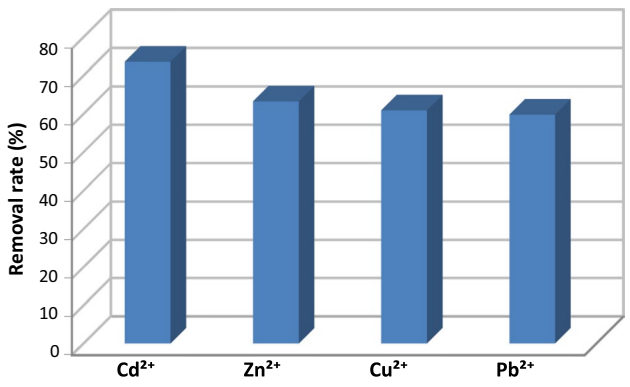

Fig. 3 Removal of heavy metals by PAC

treated by coupling infiltration-percolation and adsorption on PAC.

The results of the previous experiments show that the PAC permits satisfactory removal of heavy metals. On the other hand, the tests of analysis of the secondary effluents on the infiltration-percolation column show that the massif filtering is not effective with regard to the reduction of heavy metals. Indeed, low abatement yields were obtained, reaching 6.19, 4.55, 4.73 and 11.20%, respectively, for Zn^{2+}, Pb^{2+}, Cu^{2+} and Cd^{2+}. More efficient results are obtained by removing heavy metals at a column scale filled with Oued Mzard sand in Morocco (Aaki 2012). This shows that the efficiency of this

Table 5 Heavy metal concentrations in the secondary effluent and treated waters and removal rates

Heavy metal	Water sample	Average concentration	*R* (%)
Zn^{2+} (ppm)	E	0.0155	–
	T	0.0145	6.19
	(T + PAC)	0.0045	70.77
Pb^{2+} (ppm)	E	0.3033	–
	T	0.2886	4.55
	(T + PAC)	0.0990	66.87
Cu^{2+} (ppm)	E	0.2258	–
	T	0.2151	4.73
	(T + PAC)	0.0745	67.07
Cd^{2+} (ppm)	E	0.0087	–
	T	0.0077	11.20
	(T + PAC)	0.0018	78.42

E secondary effluent, *T* water treated by infiltration-percolation, (*T* + *PAC*) water treated by coupling infiltration-percolation with adsorption on PAC, *R* removal rate

Table 6 Variation of the heavy metal concentrations during the coupling of infiltration-percolation with adsorption on PAC

m (g)	Heavy metal	C_i (mg/l)	C_e (mg/l)	Q_e (mg/g)
45	Zn^{2+}	0.01345	0.0040	0.00021
		0.0151	0.0043	0.00024
		0.0158	0.0046	0.00025
		0.0164	0.0047	0.00026
		0.0168	0.0051	0.00026
	Pb^{2+}	0.2633	0.0806	0.00406
		0.26275	0.0861	0.00437
		0.29545	0.0925	0.00451
		0.3142	0.1072	0.00460
		0.3611	0.1294	0.00515
	Cu^{2+}	0.2011	0.0706	0.00290
		0.2161	0.0726	0.00319
		0.2288	0.0745	0.00352
		0.2338	0.0754	0.00352
		0.2396	0.0755	0.00364
	Cd^{2+}	0.0050	0.0013	0.000082
		0.0090	0.0018	0.000160
		0.00929	0.0020	0.000162
		0.00966	0.0021	0.000168
		0.0107	0.0022	0.000193

m mass of PAC, C_i initial concentration, C_e equilibrium concentration, Q_e adsorption capacity

treatment process depends on the mineralogical properties of the filter bed.

The coupling of these two processes makes it possible to improve the elimination of micropollutants by eliminating certain physico-chemical parameters which can cause saturation of the activated carbon. This increases the probability of the adsorption of these micropollutants which could compete with heavy metals on the specific surface area and at the functional sites of the PAC. The works of Guediri (2000) and Bali (2012) showed that the infiltration-percolation process allows a considerable reduction of organic matter, nitrogen pollution and suspended solids.

Simulation of adsorption equilibrium

Table 6 shows the variation in the quantity of heavy metals adsorbed at equilibrium during the coupling of infiltration-percolation and adsorption on PAC processes. The adsorption isotherms are given by the curves $Q_e = f(C_e)$ (Fig. 4).

Adsorption is a very common technique for the treatment of secondary effluents. It is a spontaneous surface phenomenon; its principle rests on the property that the solids fix certain pollutants such as heavy metals on their surfaces (Boulkrah 2008). According to Ziati et al. (2013), there is no general theory to explain the phenomenon of adsorption. However, several partial models have been proposed by different authors for static or dynamic conditions, as proposed by Jankowska et al. 1991. Indeed, the types of isotherms obtained (Fig. 4) already make it possible to draw conclusions on the interactions between the adsorbate and the adsorbent.

It has been observed according to the classification of the adsorption isotherms proposed by IUPAC (1985) that the isotherms of Zn^{2+}, Pb^{2+} and Cu^{2+} are type I isotherms. However, they are typical of monolayer adsorption and correspond to saturated micropore filling which might result in clogging. This case is analogous to chemisorption.

The isotherm of Cd^{2+} is of type II which generally corresponds to a multilayer adsorption on the open surfaces of the activated carbon. The shape of this curve corresponds to a sum of isotherms I + II, i.e., it is a micropore filling followed by a multilayer adsorption on an external surface of the adsorbent. According to Margot et al. (2011), the difference in the degree of adsorption also depends on the

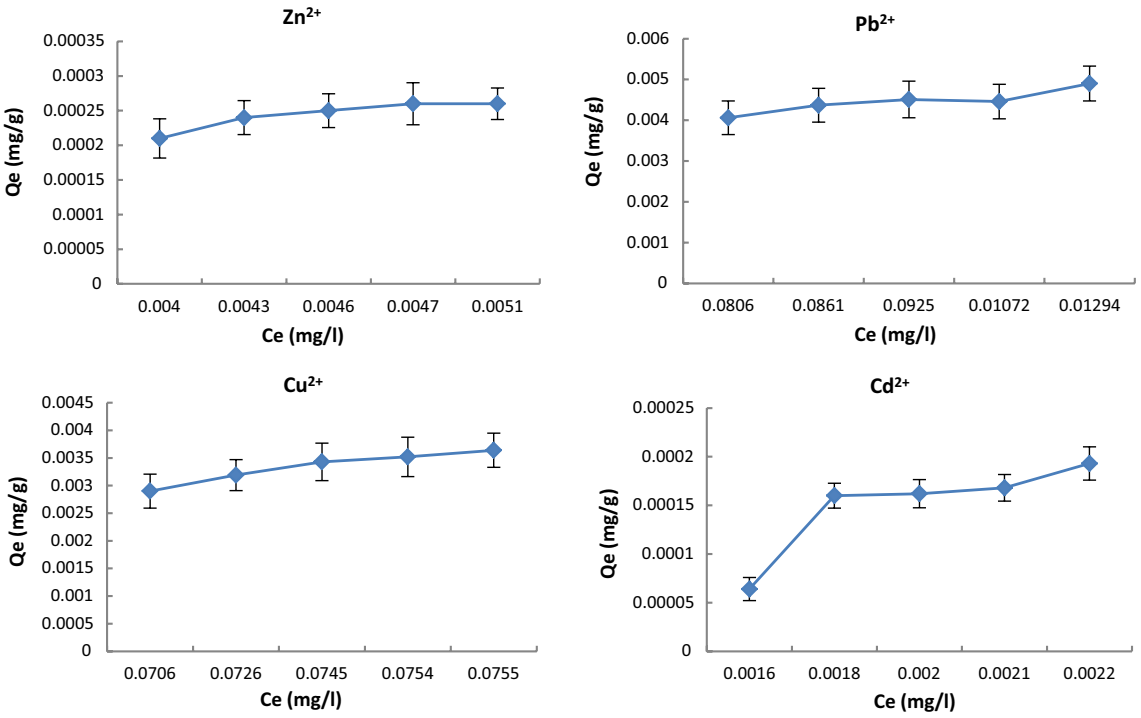

Fig. 4 Adsorption isotherms of heavy metals by the coupling of infiltration-percolation and adsorption on PAC

characteristics of the micropollutants (molecular structure, size, hydrophobicity, etc.). This can explain the difference of the PAC affinity toward heavy metals. Experimental results demonstrate that this adsorbent has the highest affinity vis-à-vis cadmium compared with the other studied heavy metals.

Conclusion

The main objective of this work was to model and classify the type of adsorption equilibrium of the heavy metals studied by coupling the infiltration-percolation and PAC adsorption processes. The experiments showed that the coupling of the two processes improves the purification of the secondary effluents. The infiltration-percolation technique, applied at a laboratory scale column according to a 4-day feeding—3-day drying schedule using a hydraulic load of 0.27 m/day, appears to be a sustainable system for removing pollutants such as organic matter and ammonium. However, it is less effective regarding heavy metal reduction. Furthermore, activated carbon is an effective adsorbent which can contribute to the satisfactory elimination of micropollutants contained in urban wastewaters thanks to its high specific surface area.

Results demonstrated that the coupling of infiltration-percolation and adsorption on PAC processes increases the abatement rate of heavy metals.

The adsorption isotherms showed that the adsorption of Zn^{2+}, Pb^{2+} and Cu^{2+} is of type I, while the Cd^{2+} has a type II isotherm. This metal has undergone a multilayer adsorption which enhances its attachment and therefore its removal from wastewater.

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