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Individual and competitive adsorption of Lead(II) and Nickel(II) ions by chemically activated carbons

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This work deals with the removal of heavy metals from wastewater which presents a major ecological problem. Recently, this has been solved by using adsorption techniques as a means to extract heavy metal ions. The present investigation was carried out on the adsorption of heavy metals from single and binary component aqueous solutions on activated carbons. The concentrations of heavy metals ions, Ni(II) and Pb(II), ranged from 10 to 100 mg.L⁻¹ and 10 to 200 mg.L⁻¹, respectively. A microporous KOH-activated carbon and a commercial mesoporous carbon were used. The first part focuses on the adsorption equilibrium of single-component metal ions solution. The KOH-activated carbon showed better performance for removal of both ions than H₃PO₄-activated carbon. The second part deals with adsorption competition between the two metals ions. The adsorption of each respective ion in a mixture was decreased compared to the single-component metal ions. Both activated adsorbents showed higher affinity for Pb(II) ions than for Ni(II) ions. The adsorption of both metal ions on adsorbents was modelled by applying Langmuir models. In addition, it has been found that the percent removal of both metallic ions is strongly concentration dependent; for low initial concentration of Ni(II) and Pb(II) it reaches 98%, while for higher initial concentrations the percent removal drops up to 78% with better performance for CKW-activated carbon.

Key words: Activated carbon, Nickel, Lead, adsorption, binary adsorption, Langmuir, affinity.

INTRODUCTION

The recovery of heavy metals from industrial aqueous effluents has gathered a great deal of attention in recent years, mainly due to the rise in environmental awareness and to the consequent severity of legislation regarding the disposal of toxic substances. The contamination by these substances affects aqueous waste streams in many industries such as metal plating, tanneries, mining operations, painting, and dyes. There are numerous processes for removing heavy metals which include chemical precipitation, membrane filtration, ion exchange,

liquid extraction or electro-dialysis (Marina et al., 2007; Sharma and Forster, 1994). These processes may be ineffective or expensive, especially at concentration in metal ion less than 100 mg/L. Adsorption methods for the removal of heavy metal ions may be an attractive alternative to physicochemical methods. The most common adsorbent materials are: alumina silica (Josefa and De Oliveira, 2003), metal hydroxides (Tiffreau et al., 1995) and activated carbon (Guo and Lua, 1999; Faur-Brasquet et al., 2002). More recently, spent coffee beans have been used for removal of indium ions from aqueous solution (Wei-Lung et al., 2012). As proved by many authors (Guo and Lua, 1999; Faur-Brasquet et al., 2002; Mellah et al., 2006; Kumar et al., 2010), removal of heavy

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metals by activated carbon is economically favourable and technically easy to handle.

Lead (Pb) and Nickel (Ni) are heavy metals which present a major risk for health and environment. Pb is one of the most ubiquitous heavy metals, causing saturnism, whereas, Ni is an oligo-element not very toxic at the natural state, but very toxic when bounded to synthesis products. A considerable number of works has been devoted to Pb and Ni ions removal by adsorption processes on activated carbon elaborated from different precursors; for instance, Kunquan and Xiaohua (2009) investigated and optimized adsorptive removal of Pb(II) by activated carbon prepared from *Spartina alterniflora*. Apipreeya et al. (2009) studied binary absorption of Cu(II) and Pb(II) to demonstrate the benefits of activated carbons prepared from eucalyptus bark. On the other hand, Kalavathy et al. (2010) have investigated Ni ions removal using activated carbon from sawdust via batch and column mode.

In this work, single and simultaneous adsorption of Pb(II) and Ni(II) on activated carbon was investigated. The adsorption study was carried out in batch mode with the same operating parameters of volume of solution, amount of activated carbon, temperature, stirring time, and pH value. Two different activated carbons were tested: a KOH-activated carbon from wood, specifically prepared at laboratory and an H₃PO₄-activated carbon, industrially produced and commercially available. The Langmuir and Freundlich models were used to predict and/or correlate mono- and multi-component equilibrium data. They were applied to the two selected metal ions in aqueous solutions.

MATERIALS AND METHODS

The adsorbent KOH-activated carbon was obtained from thermo-compressed lumps of fir-wood, following a procedure described below. The activation method required two successive steps: the precursor was firstly slowly pyrolysed in a stream of N₂ at 300°C for 1 h. The produced charcoal was then soaked in a concentrated KOH solution until an impregnation ratio of 0.75 g of KOH/1 g of initial charcoal was reached. The oven-dried impregnated product was then heated in a N₂ stream at 700°C for 2 h. The resulting material was washed with some distilled water and soaked overnight in a 0.1 M HCl solution in order to remove the remaining alkali. The material was then washed with hot distilled water (at 80°C) until the activated carbon reached a pH of 6.0 ± 0.5. The produced activated carbon by this method was designated as CKW, where C as carbon, K as potassium and W as wood. The reference activated carbon called Acticarbone CXV (CECA Company product, France) was prepared by carbonization of pine wood sawdust (*Pinus pinaster*), after impregnation with concentrated H₃PO₄.

In a previous study, the KOH-activated carbon from wood has been characterized by its spectrum in temperature programmed desorption (TPD) up to 1173K (Khezami and Capart, 2005). It has been found that the activated carbon contains carboxylic acidic groups and carboxylic anhydride groups. These functional groups play an important role in the adsorption of organic and inorganic pollutants. They confer hydrophilic properties to activated carbon by enhancing the specific interactions dipole-dipole and ion-dipole.

According to Benjamin (2002), the adsorption of cations, which apply to the present work, occurs on two different kinds of active sites: (i) the Lewis bases, O_n , and (ii) the COxHy surface groups, OH . The classical procedure used for multi-component Langmuir adsorption (Benjamin, 2002) is followed. For the case of acid COxHy sites, the capture of cations follows substitution reaction mechanisms such as described by Di et al. (2009).

Preparation of Ni(II) and Pb(II) ion solutions

Ni(II) chloride (NiCl₂) and Pb(II) nitrate (Pb(NO₃)₂) were used for the preparation of Ni(II) and Pb(II) stock solutions (1000 mg.L⁻¹) in distilled water. Experimental solutions at the desired concentration were then obtained by successive dilutions. For the pH adjustment throughout experiments, 0.1 M HNO₃ and/or 0.1 M NaOH solutions were used as necessary.

All the metal ion solutions were prepared separately as described by the European salt Producers' Association (Anonymous, 2005). For Pb²⁺ (1000 mg.L⁻¹): 1.598 g of Pb(NO₃)₂ was dissolved in about 10 ml concentrated HNO₃ [$\rho = 1.40 \text{ g ml}^{-1}$, 65% (m/m)] and diluted to 1000 ml volumetric flask with distilled water. The stock solution of Ni(II) (1000 mg L⁻¹) was prepared by dissolving 4.133 g of NiCl₂·6H₂O in distilled water. All the reagents were of analytical grade or highest purity available, and used without further purification.

Single and binary adsorption of metal ions

Monocomponent adsorption experiments were carried out in batch mode at room temperature (21 ± 1°C) and constant pH (pH 8.5) by adding 40 mg of activated carbon (CKW or Acticarbone) to 100 cm³ of Ni(II) and Pb(II) metal ions solutions in a 250 cm³ Erlenmeyer. The initial concentration of Ni(II) and Pb(II) ions ranged from 5 to 100 mg.L⁻¹ and 5 to 200 mg.L⁻¹, respectively. Binary adsorption experiments of Ni(II) and Pb(II) metal ions were performed under the same operating conditions. Initial concentrations of both the metal ions ranged from 5 to 50 mg.L⁻¹.

In all the experiments, the pH was fixed to 8.5. Several flasks were placed in a thermostatic bath and individually stirred at 450 rpm. After 12 h of contact, samples were withdrawn from each flask and filtered using 0.45 µm cellulose acetate syringe filters. The residual concentration of Ni(II) and Pb(II) were spectrophotometrically measured at their respective maximum wavelength of 445 and 515 nm, according to the chemical supplier standard method (included with the chemicals as supplied by Merck®). This method consists in measuring the absorbance of a Pb(II) sample of filtrate (5 ml) that contains a primary lead dithizonate formed by dithizone in presence of cyanide at a pH 7 to 9. The filtrate is then extracted by carbon tetrachloride to give a sensitive pink-red colour. The Ni(II) ions form a brown-red colour after oxidation by diacetyldioxime in alkali medium. All the chemicals supplied by Merck®, have an analytical reagent grade. All the experiments were duplicated and the mean values reported. The maximum deviation was 3%.

Equilibrium models

The equilibrium adsorption isotherm is vital to the design of adsorption systems, and its shape provides information about homogeneity and heterogeneity of the adsorbent surface. Moreover, the correlation of the equilibrium data with either theoretical or empirical equations is essential to practical operation. The equation of Langmuir is the most widely applied for modelling equilibrium data of adsorption. It assumes that there is no interaction between the adsorbed molecules and adsorption is

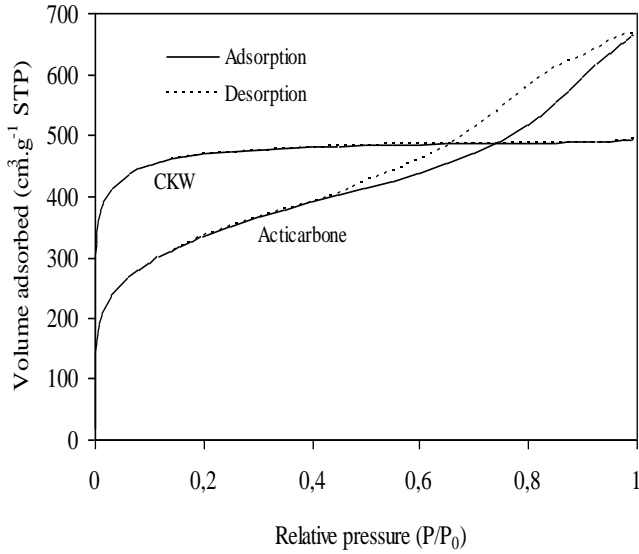


Figure 1. Adsorption-desorption isotherms of N₂ at 77K on CKW and Acticarbone activated carbon.

localized in a monolayer (that is, onto planar surfaces with a finite number of identical adsorption sites, homogeneously distributed on the adsorbent surface). The Langmuir isotherm is represented by the following Equation 1:

$$q_e = \frac{Q_0 \cdot b_i \cdot C_e}{1 + b_i \cdot C_e} \quad (1)$$

Where C_e is the equilibrium concentration of solute in the bulk solution ($\text{mg}\cdot\text{L}^{-1}$), Q_0 is the ultimate solid phase concentration corresponding to the complete monolayer coverage of adsorption sites, and b is the constant related to the free energy of adsorption. Values of Q_0 and b_i can be graphically determined from the linearized Langmuir equation, where the slope is $1/Q_0$ and the intercept $1/Q_0 \cdot b_i$. From the corresponding Langmuir parameters, the dimensionless parameter R_L or "separation factor" was calculated. This factor is defined as follows:

$$R_L = \frac{1}{1 + b_i \cdot C_0} \quad (2)$$

Where C_0 is the initial concentration of Ni(II) and Pb(II). Thus, R_L is a positive number whose magnitude determines the feasibility of the sorption process. The process is irreversible if $R_L = 0$, favorable if $R_L < 1$, linear if $R_L = 1$, and unfavorable if $R_L > 1$.

Actually, wastewaters do not contain only one ion but many ions of pollutants, yielding difficult the description of the pollutants adsorption from waste streams. Interference and competition phenomena for adsorption sites occur when several ions are present and lead to a more complex mathematical formulation. Therefore, various isotherm models have been proposed to describe adsorption equilibrium and competition between components for such a system. A possible model for two metal systems is given by a modified Langmuir model based on the same hypothesis as for a single component. The modified Langmuir model contains interaction terms η_i which characterize each metal ion and depend on the concentration of the other ion in solution

(Pagnanelli et al., 2004). The modified Langmuir model is written as:

$$q_1 = \frac{Q_1^0 \frac{b_1}{\eta_1} C_1}{1 + \frac{b_1}{\eta_1} C_1 + \frac{b_2}{\eta_2} C_2} \quad (4)$$

$$q_2 = \frac{Q_2^0 \frac{b_2}{\eta_2} C_2}{1 + \frac{b_1}{\eta_1} C_1 + \frac{b_2}{\eta_2} C_2} \quad (5)$$

In Equations 4 and 5, for the first and the second metallic ion in solution at equilibrium, b_1 and b_2 , are the respective individual Langmuir adsorption constants, C_1 and C_2 are the residual concentrations ($\text{mg}\cdot\text{L}^{-1}$), q_1 and q_2 are the adsorbed quantities per gram of activated carbon ($\text{mg}\cdot\text{g}^{-1}$), Q_1^0 and Q_2^0 are the individual maximum specific quantities (obtained by adsorption data of binary system), η_1 and η_2 are the Langmuir correction coefficient of the first and the second metallic ion estimated from competitive adsorption data. For binary mixture, Equations 4 and 5 can be solved simultaneously to obtain the multi-component Langmuir correction coefficients η_1 and η_2 of the first and the second components, respectively.

The extent of metallic ions removal (in %) can be deduced from the initial concentration (C_0) and the equilibrium one (C_e) as follows:

$$R(\%) = 100 \times \frac{C_0 - C_e}{C_e} \quad (6)$$

RESULTS AND DISCUSSION

Adsorption of nitrogen

Based on the Brunauer's classification of sorption isotherms (Rouquerol et al., 1999), the adsorption isotherm of N₂ at 77K for KOH-activated carbon (CKW) is of Type I (Figure 1). In addition, the absence of hysteresis indicates that the internal structure of the activated carbon is mainly micro-porous with a narrow distribution of pore sizes. Whereas, a H₃PO₄-activated carbon such as Acticarbone CXV, practically exhibits a type IV sorption isotherm (Rouquerol et al., 1999), which is specific of a mesopores structure (Table 1).

The H4 type hysteresis given by Figure 1 indicates a pores shape more flat than cylindrical. Issa and Teresa (2000) reported similar effects of the alkaline and acidic activating agents, on the aspects of sorption isotherms.

The main physical characteristics of CKW and Acticarbone powders are shown in Table 1. It can be noted from this table that the KOH-activated carbons average pores diameter is just under 20 Å, thus, confirming the microporous structure described by Figure 1. While, for Acticarbone CXV, the average pores diameter is about 33 Å and a high mesopores surface

Table 1. Physical characteristics of CKW and Acticarbon powders.

Activated carbon	Micropore surface area (m ² .g ⁻¹)	Mesopore surface area (m ² .g ⁻¹)	Micropore volume (cm ³ .g)	Average pore diameter (Å)
CKW	988	267	0.596	19.34
Acticarbon	230	980	0.091	33.26

Table 2. Langmuir adsorption constants of Ni and Pb in single and binary solution at 21°C and pH = 8.5.

Activated carbon	ions	Q ₀	b _i	R _L	Q ₀ ⁱ	η _i
CKW	Ni(II)	160.9	0.323	0.058	139.1	2.21
	Pb(II)	355.3	0.840	0.023	214.7	1.72
Acticarbon	Ni(II)	109.6	0.342	0.076	96.5	2.67
	Pb(II)	230.7	0.476	0.403	170.5	1.37

area (980 m².g⁻¹). Guo and Lua (1999) prepared an activated carbon from oil-palm stone and shown that the KOH-activated carbon is essentially microporous with a high surface area of pores, while the H₂SO₄-activated carbon has a more mesoporous structure.

Effect of initial metal ions concentrations

The effect of the initial concentration on the Ni(II) and Pb(II) is investigated in the range of 10 to 100 mg.L⁻¹ and 10 to 200 mg.L⁻¹, respectively, in the case of individual adsorption. If the amount in CKW in the suspension is equal to 0.4 g.L⁻¹, 95 and 98% of the initial Ni(II) and Pb(II), respectively, with a concentration of 10 mg.L⁻¹ are removed. Whereas, if the initial concentration is about 100 mg.L⁻¹ for Ni(II) and 200 mg.L⁻¹ for Pb(II), the amount removed drops to 79 and 77%, respectively. Using Acticarbon in amount of 0.6 g.L⁻¹, the amounts of Ni(II) and Pb(II), removed are, respectively, 97 and 98% for initial concentrations of 10 mg.L⁻¹. Moreover, when the initial concentration is about 100 mg.L⁻¹ for Ni(II) and 200 mg.L⁻¹ for Pb(II), the amount removed drops to 77%, respectively. Therefore, the uptake of Ni(II) and Pb(II) is strongly concentration-dependant and it can be seen that this uptake is higher for CKW than it is for Acticarbon, in particular for higher initial concentration. This higher uptake is probably due to the microporous structure of KOH-activated carbon in contrast with that of acid-activated carbon whose volume of mesopores is relatively larger (Table 1).

The effect of the pH on adsorption

The value of pH was fixed after a literature examination

(Faur-Brasquet et al., 2002; Rao et al., 2000; Bayat, 2002; El-Ashtoukhy et al., 2008). According to Faur-Brasquet et al. (2002), who have investigated the adsorption of heavy metals (Copper, Nickel and Lead) on activated carbon, the Ni(II) adsorption is optimal in a pH range between 4 and 9. This result is explained by the formation of the complex Ni(OH)⁺ in this interval of pH, as it was corroborated by Rao et al. (2002), when studying the capture of Cr(VI) and Ni(II) ions from aqueous solution by using bagasse and fly ash as sorbent materials. These authors have reported that the adsorption of Ni(II) is maximal at pH = 8 and that at pH ≥10, Nickel precipitates. Moreover, Bayat (2002) showed that the adsorption of the Ni(II) ions by ashes is maximal at pH ≥8. El-Ashtoukhy et al. (2008) reported that the maximal removal of Pb(II) ions on activated carbon was observed in a range of pH between 5.6 and 7.6. Faur-Brasquet et al. (2002) showed that the precipitation zone of Pb corresponds to a range of pH from 7 to 8. As a conclusion, on the one hand, Ni precipitates at pH ≥10 and its uptake is optimal at pH ≥8, while on the other hand, Pb precipitates in a pH range between 7 and 8 and its uptake is large at alkaline pH. For these reasons the choice of pH = 8.5 seems to be a good compromise for the binary adsorption of Ni and Pb.

The linearized Langmuir isotherms of Ni(II) and Pb(II) in single solutions are drawn and the estimated model parameters (Q₀, b) with correlation coefficient (R²) are shown in Table 2. The curves of adsorption isotherms present a plateau for large metallic ion concentrations, and their shape is characteristic of type L isotherm (Figure 2a and b).

For both the tested adsorbents, it is obvious from Table 2 that the maximal values of adsorption capacity (Q₀) for Pb(II) ions were always higher than those for Ni(II) ions. For removing the two tested ions, the KOH-activated

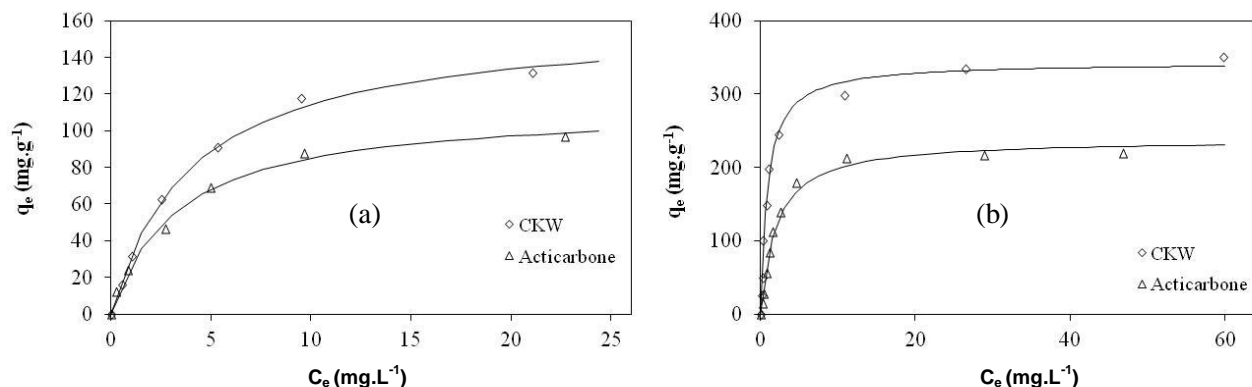


Figure 2. Langmuir adsorption isotherms of (a) Ni(II) and (b) Pb(II) separately on activated carbons (a) CKW and (b) Acticarbone at 25°C and at pH 8.5.

carbon CKW showed a better efficiency than the H_3PO_4 -activated carbon (Acticarbon), commercially available. For example, the adsorption capacity of Pb(II) ions was about 355 mg.g^{-1} with CKW and only 230.7 mg.g^{-1} with Acticarbon CXV. These differences could be explained either by the chemical nature of the functional groups in each adsorbent or by their porous structure (microporous and mesoporous). Thus, it was demonstrated in a previous work (Khezami and Capart, 2005) that KOH-activated carbon has a higher content in COOH surface functional groups. These groups are mainly involved for the removal of metallic ions, as proved by Jia and Thomas (2000).

This result is similar to that obtained by Faur-Brasquet et al. (2002) who emphasized the higher adsorption capacity of microporous activated carbon when compared to mesoporous activated carbon, for the uptake of Cu(II) and Pb(II) ions. The reported values of Q_0 in the literature vary in a wide range according to the experimental conditions, the nature of sorbent, and the ionic species; thus, Kadirvelu et al. (2002) tested the performance of a ZnCl_2 -activated carbon to eliminate Ni(II) from aqueous solution at different pH values, and they found a maximum Q_0 value of 54 mg.g^{-1} . When studying the adsorption of Pb(II) on a modified activated carbon-containing alginate bead (AC-AB), Hyun et al. (2007) found an adsorption capacity Q_0 of about 333 mg.g^{-1} .

The values of dimensionless separation parameter R_L are shown in Table 2 for Ni and Pb, respectively. In all cases, the R_L values evaluated from experimental data were found less than unity, thus attesting a favourable adsorption of the two metals on the adsorbents. For the linearized data of the Langmuir model, the values of R^2 are close to 1, thus attesting the goodness-of-fit to the isotherm model.

The performances of the two adsorbents used for the binary adsorption of Ni(II) and Pb(II) ions were drawn by the modified Langmuir model. The main advantage of this

model is that it can represent/predict the binary adsorption isotherms using parameters obtained from single component isotherms. In this case, the model without the correction coefficient η_i reproduce the isothermal data of single metal systems (Figure is not showed in this paper). Moreover, the predictive model containing η_i and obtained by fitting to binary data represented by the 3D plot of Ni-Pb adsorption (Figure 3), cannot reproduce single metal equilibrium data especially for Ni and gives a binary representation reflecting larger Pb affinity with respect to the adsorbent material.

Table 2 shows the various parameters obtained for this binary mixture. From the data presented in Table 2, it is clear that even in the competitive adsorption situation, CKW has a higher capacity than Acticarbone to remove both metal ions. This is confirmed by the Langmuir adsorption capacities, which, in all cases, are higher for CKW than for Acticarbone, for both the single (Q_0) and competitive (Q'_0) adsorption of the chosen metals. As shown above and in a previous study, with respect to Acticarbone, CKW has a higher microporosity and a better development of chemical surface (Khezami and Capart, 2005).

As a first approach, one can note that for a given metal, the adsorption capacity Q'_0 is definitely lower for co-adsorption than for simple adsorption. The competition between ionic species toward adsorption can be emphasized by simply considering the ratio Q'_0/Q_0 as did by Otero et al. (2009), for the removal of heavy metals by sewage sludge. If $Q'_0/Q_0 > 1$, the adsorption is enhanced by the presence of another metal ion. If $Q'_0/Q_0 = 1$, there is no observable net interaction, and finally when $Q'_0/Q_0 < 1$, adsorption is suppressed by the presence of the competitive metal ion. Thus, in the present study, the adsorbed amount of a given metal Ni or Pb from of a binary solution is less than the adsorbed amount of the same metal from a single component solution. These

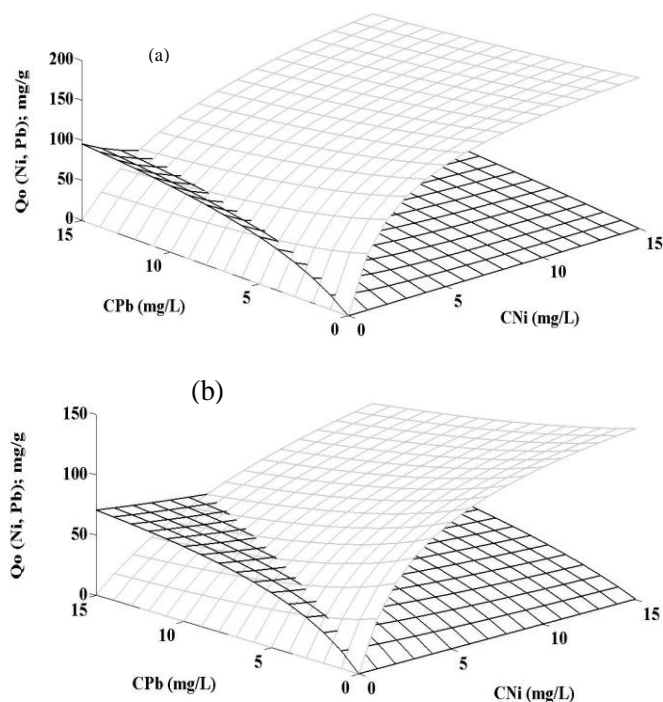


Figure 3. Adsorption onto (a) CKW and (b) Acticarbon activated carbons of Ni-Pb binary solution (21°C, pH = 8.5) by modified Langmuir model Equations 4 and 5.

results are in concordance with other investigations done on single and binary adsorption of heavy metals (Otero et al., 2009).

One type of metal ion affects the uptake of another one, and the total uptake in a multi-components system is slightly lower than the uptake in a corresponding single system. Actually, the preference or the affinity for Ni(II) or Pb(II) of the two tested activated carbons can be expressed by the separation factor of Helfferich (1995). This factor is defined as follows:

$$\alpha_{Pb}^{Ni} = \frac{q_{Ni} C_{Pb}}{q_{Pb} C_{Ni}} \quad (8)$$

Where, C_{Ni} , C_{Pb} , and q_{Ni} , q_{Pb} are the respective equilibrium concentration and adsorption capacities of Ni(II) and Pb(II) in solution. If Ni(II) has more affinity for the adsorbent, the separation factor α_{Pb}^{Ni} is higher than 1, and vice versa. This factor was calculated for the two adsorbents, and the obtained values were 0.385 and 0.621 for CKW and Acticarbon, respectively. This result clearly shows that between the two tested metal ions, Pb(II) has the larger affinity for the activated carbons, moreover alkali activated carbon CKW revealed to be more effective than the acid activated carbon Acticarbon to eliminate from aqueous solutions heavy metals ions

such as Ni(II), Pb(II) and Cr(VI) (Khezami and Capart, 2005). This finding can be explained by the fact that the functional groups on the surface of CKW and Acticarbon have a relatively stronger affinity for Pb^{2+} than Ni^{2+} .

Conclusion

In order to examine the availability and effectiveness of a KOH-activated carbon prepared from wood for the removal of heavy metals from aqueous solutions, the heavy metals removal capacity was observed in comparison with that of a commercial activated carbon. The KOH-activated carbon is an effective adsorbent for removal of Pb and Ni ions from single- and binary-component aqueous solutions. In fact, the chemical nature of the functions groups of each adsorbent and their respective porous structure might be the origin of this result. The ultimate adsorption capacity (Q_0) is definitely lower in the case of binary-adsorption than in the case of single-adsorption, regardless of the adsorbent. Moreover, The study indicates that the removal of these heavy metals is selective, with Pb(II) being removed preferentially to Ni(II).

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