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MULTI-METAL SORPTION BY SPENT ANIMAL BONES

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ABSTRACT

The multi-component metal sorption by animal bones was examined using single, binary, and ternary systems composed of Cu^{2+}, Zn^{2+}, and Ni^{2+}. Four isotherm models, namely Freundlich, Langmuir, Sips, and Ideal Adsorption Solution Theory (IAST) were used in this study to predict the equilibrium uptake for Cu^{2+}, Zn^{2+}, and Ni^{2+} in binary solutions using the single adsorption constants, obtained by the single isotherm experimental data. Sorption was suppressed by the presence of other metal ions in the binary or ternary sorption processes.

The Langmuir, Freundlich, and Sips isotherm models were found to represent the experimental data of a single isotherm sorption process. Whereas, the predictions of binary sorption isotherm systems, Cu^{2+}–Ni^{2+}, Zn^{2+}–Cu^{2+}, Ni^{2+}–Zn^{2+}, showed good agreement with experimental data when using all models except Langmuir.

Key Words: Animal bones; Copper; Multi-metal; Nickel; Sorption; Zinc

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INTRODUCTION

The presence of heavy metals in wastewater is one of the most important problems in environmental engineering today, due to their high toxicity and cumulative character. Industrial processes that generate effluents with high heavy metals content are both numerous and diverse: metal processing, finishing, and plating industries are obvious sources of metal wastes (1).

Copper, zinc, and nickel are among the heavy metals that affect the environment. Many studies have shown that these metals are harmful even at low concentrations. Copper and zinc, in particular, have the capability to induce the protein *Patella vulgata* in marine organisms to bioaccumulate more toxic heavy metals (2). Therefore, their presence in wastewater is as important as the presence of other more toxic heavy metals. However, these metals have many applications in the industrial life. For example, copper is known as an excellent conductor of electricity and is widely used in the electro-industry. It is also utilized as an analytical reagent (in the form of salts or ions), in paints for ships' bottoms, and in electroplating. However, it acts as an irritant to the skin, causing itching and dermatitis, and may cause keratinization of the hands and soles of the feet (3).

Therefore, it is necessary to decrease the concentration of heavy metals to their permissible limits before their discharge into the environment. The use of adsorbents, e.g., activated carbon and ion-exchange resins, to remove trace metals from aqueous system has been widely investigated (4–6). However, because of high capital and regeneration costs of activated carbon and ion exchange resins (7), researchers are encouraged to look for other types of adsorbents. Considerable research has involved materials of biological origin and many forms of biomass have been shown to be effective for the removal of heavy metals (8–11). These materials are known as “biosorbents”. The uptake of metals by these materials was attributed to their constituents of proteins, carbohydrates, and lignin that contain functional groups, such as carboxyl, hydroxyl, and amine groups, that are responsible for metal sorption (12). Therefore, biosorption is a physio-chemical binding of a substance, sorbate, to a biological material.

Industrial effluents often contain more than one metal. Therefore, it is necessary to study the sorption from a mixture of metal ions, since the degree of metal removal from their solutions depends on the multi-metal competition interactions in solution with the sorbent material. Therefore, it is important to understand the behavior of this mixture in the solution and to explore the effect of the concentration of one metal ion on the uptake of other metal ions. This can be achieved by investigating the equilibrium isotherm of multi-component systems.

In this work, sorptions of metals from solutions containing two or three various metals by animal bones were studied. Four isotherm models, namely

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Freundlich, Langmuir, Sips, and Ideal Adsorption Solution Theory (IAST) were used to predict the equilibrium uptake for Cu^{2+}, Zn^{2+}, and Ni^{2+} in the binary solutions using the single adsorption constants.

THEORY

Four isotherm models, namely Freundlich, Langmuir, Sips, and IAST, were used in this work.

Freundlich Model

This model is derived from the Gibbs adsorption equation combined with a mathematical description of the free energy of the surface (13). Freundlich model describes adsorption in terms of the sorbate concentration and is given by

$$q_{\mathrm{e}} = k_{\mathrm{F}} C^{1/n} \tag{1}$$

where k_{F} is a Freundlich constant related to the sorption capacity, and $1/n$ is a Freundlich constant related to the sorption intensity.

In addition, the Freundlich model can be extended to describe the multicomponent adsorption process. For a binary system, the expression for q_1, the uptake of species 1, in this binary system (14) is:

$$q_1 = \frac{n\left(\frac{k_{\mathrm{F1}}}{n_1}\right)^{1/n_1} C_1}{\left[\left(\frac{k_{\mathrm{F1}}}{n_1}\right)^{1/n_1} C_1 + \left(\frac{k_{\mathrm{F2}}}{n_2}\right)^{1/n_2} C_2\right]^{1-n}} + \Delta F_2 \tag{2}$$

where

$$\Delta F_2 = (n_1 - n_2) \times \frac{\left(\frac{k_{\mathrm{F1}}}{n_1}\right)^{1/n_1} C_1 \left(\frac{k_{\mathrm{F2}}}{n_2}\right)^{1/n_2} C_2}{\left[\left(\frac{k_{\mathrm{F1}}}{n_1}\right)^{1/n_1} C_1 + \left(\frac{k_{\mathrm{F2}}}{n_2}\right)^{1/n_2} C_2\right]^{2-n}} \ln\left[\left(\frac{k_{\mathrm{F1}}}{n_1}\right)^{1/n_1} C_1 \left(\frac{k_{\mathrm{F2}}}{n_2}\right)^{1/n_2} C_2\right] \tag{3}$$

$$n = \frac{n_1\left(\frac{k_{\mathrm{F1}}}{n_1}\right)^{1/n_1} C_1 + n_2\left(\frac{k_{\mathrm{F2}}}{n_2}\right)^{1/n_2} C_2}{\left(\frac{k_{\mathrm{F1}}}{n_1}\right)^{1/n_1} C_1 + \left(\frac{k_{\mathrm{F2}}}{n_2}\right)^{1/n_2} C_2} \tag{4}$$

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where C_1 and C_2 are the equilibrium concentrations for species 1 and 2, respectively. The parameters k_{F1} and k_{F2}, n_1, and n_2 are Freundlich constants, which can be obtained from single adsorption isotherm systems using SIGMAPLOT 5.0 package (Jandel Scientific, Cort Madera, CA), by means of non-linear regression analysis.

Langmuir Model

The Langmuir model was originally developed to describe and quantify sorption on a set of distinct localized adsorption sites, and it has been used to describe both physical and chemical adsorption. The Langmuir equation relates solid phase adsorbate concentration (q_e), the uptake, to the equilibrium liquid concentration (C) as follows:

$$q_e = \frac{k_L bC}{1 + bC} \tag{5}$$

where k_L and b are the Langmuir constants, representing the maximum adsorption capacity for the solid phase loading, and the energy constant related to the heat of adsorption, respectively.

The above model can be extended to describe a multicomponent adsorption system. In this case, the Langmuir model (often called extended-Langmuir) can be written as:

$$q_i = \frac{k_{Li} b_i C_i}{1 + \sum_{k=1}^{N} b_k C_k} \tag{6}$$

where q_i is the uptake of component i in the multicomponent system and C_k ($k = 1, 2, \ldots, N$; N is the number of components) is the equilibrium concentration of each component in the system and k_{Li} and b_i are Langmuir constants of species i. The Langmuir parameters were also calculated by using SIGMAPLOT 5.0 package.

Sips Model

The Sips model is another empirical model for the representation of equilibrium adsorption data. It is a combination of Langmuir and Freundlich isotherm type models. The Sips model takes the following form for single metal equilibrium data (14):

$$q = \frac{k_S (bC)^{1/n}}{1 + (bC)^{1/n}} \tag{7}$$

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Unlike the other-mentioned models, this model contains three parameters: k_S, b, and $1/n$, which can be evaluated by using SIGMAPLOT 5.0 package. The Sips model can be extended to describe the multicomponent adsorption equilibrium data, which can be written in different forms; one of these is

$$q_i = \frac{k_{Si} b_i C_i \left(\sum_{k=1}^{N} b_k C_k\right)^{1/n_i - 1}}{1 + \left(\sum_{k=1}^{N} b_k C_k\right)^{1/n_i}} \tag{8}$$

In this case, k_{Si}, n_i, and b_i are the single-metal Sips constants of species i as obtained from Eq. (7). It is important to point out that even if single component isotherm experimental data are well represented by the Sips model, Eq. (7), this does not necessarily imply that Eq. (8) will give a good representation of the multicomponent adsorption experimental data (14).

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Ideal Adsorption Solution Theory (IAST)

The IAST is another model that can be used to predict the multicomponent adsorption isotherms using single metal equilibrium data. It is based on the assumption that the solution is ideal, i.e., if there is no adsorbed–adsorbent interaction. With the single-metal data described by the Freundlich model, this model has been successfully used to describe multicomponent adsorption of competing compounds (15). In this case, the model can be expressed as:

$$C_i = \frac{q_i}{\sum_{j=1}^{N} q_j} \left[\frac{\sum_{j=1}^{N} n_j q_j}{n_i k_{Fi}}\right]^{n_i} \tag{9}$$

where N is the number of components in the system, n and k_F are the single-metal Freundlich constants of species i. In the case of a binary system, Eq. (9) can be written separately for each component. The resulted equations are to be solved simultaneously to obtain q_1 and q_2 for given equilibrium bisolute concentrations, C_1 and C_2. The solution procedure was adopted from Weber and Smith (16). To illustrate, Eq. (9) can be rewritten as:

$$\mathbf{F} = F(q_i) = C_i - \frac{q_i}{\sum_{j=1}^{N} q_j} \left[\frac{\sum_{j=1}^{N} n_j q_j}{n_i k_{Fi}}\right]^{n_i} \tag{10}$$

The roots of this equation can be obtained by means of iterative Newton–Raphson technique, according to

$$\mathbf{q}_{\text{new}} = \mathbf{q}_{\text{old}} - \mathbf{J}_{\text{old}}^{-1} \mathbf{F}_{\text{old}} \tag{11}$$

where $\mathbf{J}_{\text{old}}^{-1}$ is the inverse of Jacobian matrix, a square matrix with elements

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$\partial F_i(q)/\partial q_k$, and i and k are row and column positions in the matrix, respectively. The diagonal elements of the Jacobian matrix are given by

$$-\frac{1}{\sum q}\left[\frac{\sum nq}{n_i k_{\mathrm{F}i}}\right]^{n_i} + \frac{q_i}{(\sum q)^2}\left[\frac{\sum nq}{n_i k_{\mathrm{F}i}}\right]^{n_i} - \frac{q n_i^2}{\sum q}\frac{[\sum nq]^{(n_i-1)}}{[n_i k_{\mathrm{F}i}]^{n_i}} \tag{12}$$

while the off-diagonal elements are given by

$$-\frac{q}{(\sum q)^2}\left[\frac{\sum nq}{n_i k_{\mathrm{F}i}}\right]^{n_i} - \frac{q n_k n_i}{\sum q}\frac{[\sum nq]^{(n_i-1)}}{[n_i k_{\mathrm{F}i}]^{n_i}} \tag{13}$$

The above procedure was programmed using the package MATLAB (Version 5.2).

MATERIALS AND METHODS

Sorbent

Animal bones (from cows) were collected from butchers' shops. All the attached meat and fat were removed and cleaned from the bones. After that, the bones were washed several times with tap water and certain washing reagent, to remove entirely the meat, grease, and medulla from the bones, and left in open air for several days to get rid of the odors. Then, they were transferred to the oven at 80°C for 24 hr. The dry bones were crushed and milled into different particle sizes in the range 0.71–2.0 mm and were later used as sorbent in the sorption tests.

Batch Sorption Experiments

In batch process, 50 mL of solution, either single metal or a mixture, was transferred into plastic bottles. The mixture was prepared by solubilizing a combination of either Cu^{2+}–Ni^{2+}, Zn^{2+}–Cu^{2+}, or Ni^{2+}–Zn^{2+} or Cu^{2+}–Zn^{2+}–Ni^{2+} system, with each metal in the concentration range 0–100 ppm. Copper, zinc, and nickel were in the form of $CuSO_4{\cdot}5H_2O$, $ZnSO_4{\cdot}7H_2O$, and $Ni(NO_3)_2{\cdot}6H_2O$, respectively. All the chemicals are of analytical grade (BDH Chemicals Ltd, Poole, England). The pH of the solutions was adjusted between 4.5–5.0 by adding droplets of either 0.1 *M* HCl or 0.1 *M* NaCl solution. After that, sorbent was added to make its concentration 4 mg/mL in the suspension. A water shaker (Kottermann, Germany) was used to agitate the mixture, at 23°C, and the bottles were removed once equilibrium was achieved, by allowing sufficient contact time for the sorption process (about 24 hr). The sorbent was separated from the samples by centrifugation (3000×g, 10 min) and the upper layer was analyzed for the metals under consideration using flame atomic absorption spectrophotometer (Spectro

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AA10, Varian, Germany) using the wavelengths 218.2, 307.6, 341.5 for Cu, Zn, and Ni, respectively. Manufacturer specifications were followed to avoid any cross-interference in determining metal ion concentrations by the AA10.

When blank trials with sorbent addition were performed, neither precipitate nor metal ions adsorbed to the wall of the bottle with the tested metals under the experimental conditions. Each experiment was carried out in duplicate and the average results are presented in this work.

RESULTS AND DISCUSSION

Single-Metal Sorption

The experimental data of the single-metal sorption isotherms for Cu^{2+}, Zn^{2+}, and Ni^{2+} are shown in Figs. 1–3, respectively. The isotherm models, Eqs.

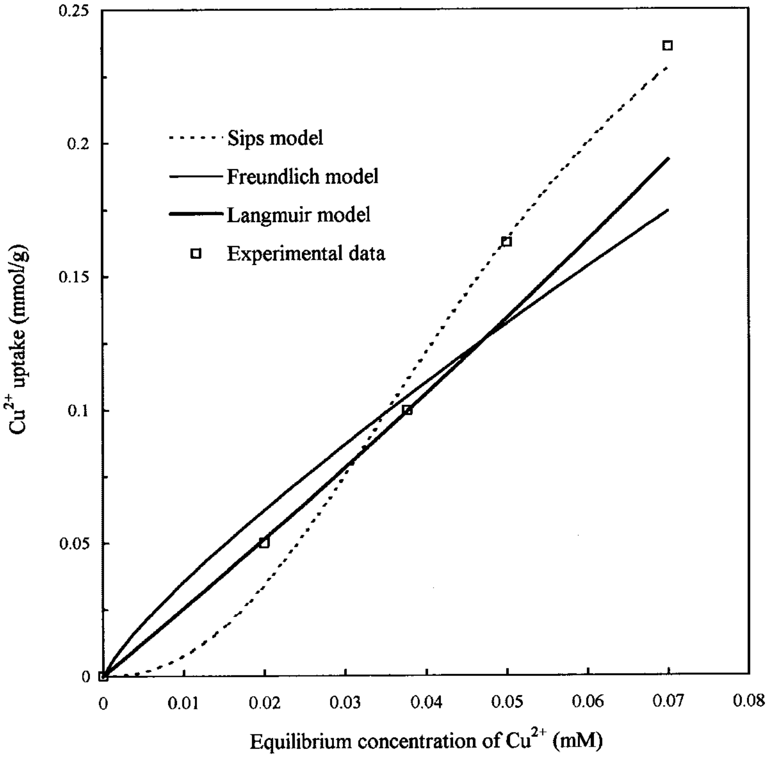

Figure 1. Sorption isotherm of Cu^{2+} by animal bones using different isotherm models.

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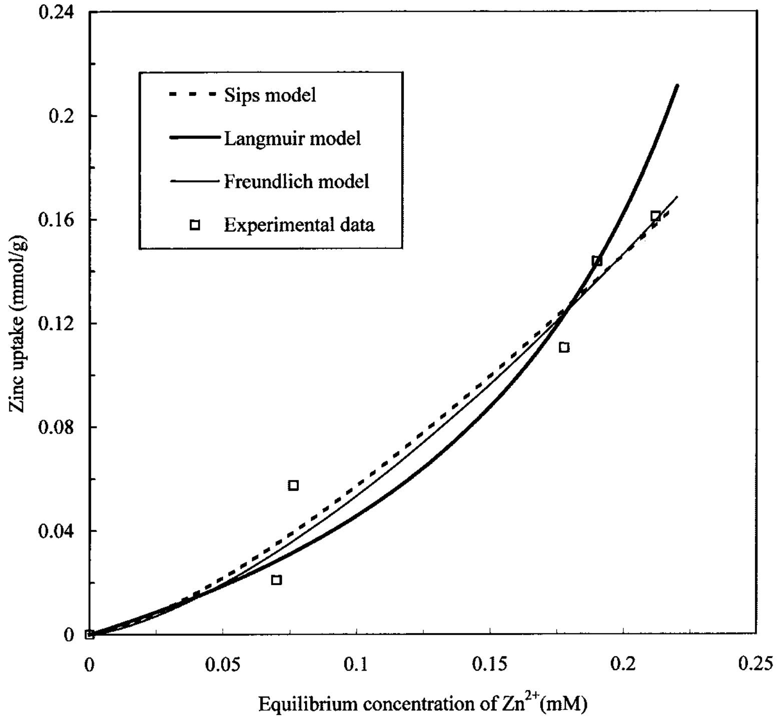

Figure 2. Sorption isotherm of Zn^{2+} by animal bones using different isotherm models.

(1), (5) and (7), were used to fit the equilibrium data of each metal. The software package SIGMAPLOT 5.0 was used to the regressed parameters for these models, using the experimental data for each of the three metal ions, by the non-linear least-square regression (Table1). These results indicate that an increase in the metal ion concentration resulted in an increase in the amount of ions adsorbed. The capacity of the bone for these metals, based on Freundlich or Sips models, followed the order $Cu^{2+} = Zn^{2+} > Ni^{2+}$. It is seen (Figs. 1–3) that the three models fit the experimental data reasonably well. The values of R^2, which is a measure of the goodness-of-fit, confirm the good representation of the experimental data by these models for the case of Cu^{2+} and Ni^{2+} isotherms and poor fitting of the Langmuir model to Zn^{2+} uptake.

The applicability of the Langmuir model to the experimental data of Figs. 1–3 indicates monolayer coverage on the bone surface of each of these metals, individually. The apparent dissociation constant for the sorption system (K_d) represents the ratio of the desorption rate constant to the adsorption rate constant. This constant is the inverse of the Langmuir constant b. The K_d values, which can

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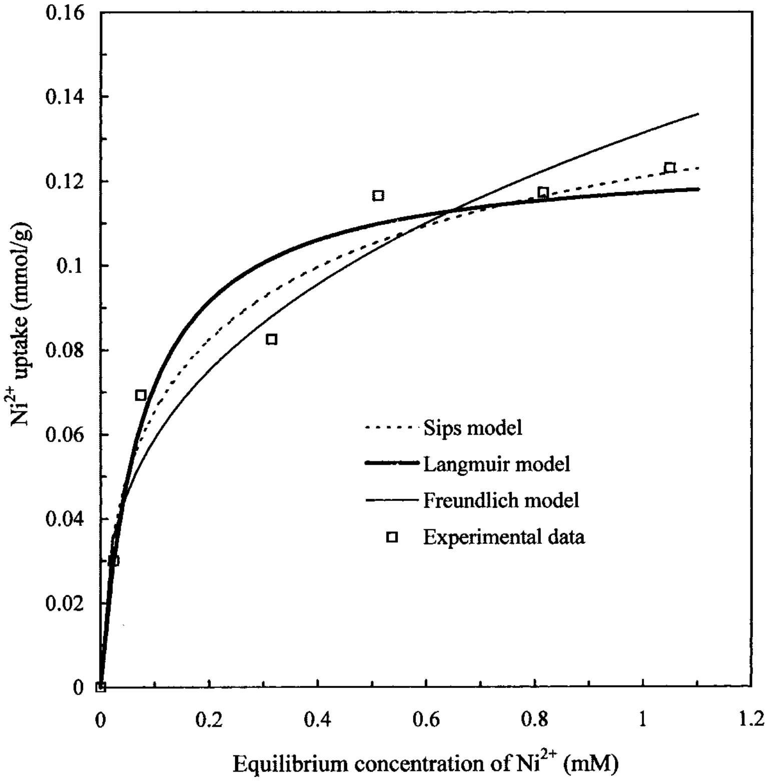

Figure 3. Sorption isotherm of Ni^{2+} by animal bones using different isotherm models.

be concluded from Table 1, indicate that Ni^{2+} has a much higher 'net' desorption rate than Zn^{2+} and Cu^{2+}.

Binary-Metal Sorption

Binary system equilibrium was considered in this study using the three binary systems Cu^{2+}–Ni^{2+}, Zn^{2+}–Cu^{2+}, and Ni^{2+}–Zn^{2+}. Experimental data for binary adsorption for the systems Cu^{2+}–Ni^{2+}, Cu^{2+}–Zn^{2+}, and Ni^{2+}–Zn^{2+} are shown in Figs. 4–6, respectively. These figures show the relationship between bound-metal concentration as mole fraction (y) and free-metal concentration in liquid phase (x). These figures also show the predicted binary isotherm data using the four mentioned models. The mole fractions y and x were calculated as

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Table 1. Model Isotherm Parameters for the Single Metal Sorption Systems

	Langmuir Model			Freundlich Model			Sips Model			
Sorbate	k_L (mmol/g)	b $(mM)^{-1}$	R^2	k_F	n	R^2	k_s (mmol/g)	b $(mM)^{-1}$	n	R^2
Cu	1.87	1.3	0.98	1.542	1.22	0.94	1.95	19.3	0.43	0.98
Zn	0.126	3.0	0.88	1.524	0.68	0.97	1.874	0.88	0.7	0.99
Ni	0.105	13.2	0.99	0.132	2.88	0.95	0.177	3.91	1.78	0.99

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follows:

$$x_i = \frac{C_i}{\sum_{i=1}^{n} C_i} \tag{14}$$

$$y_i = \frac{q_i}{\sum_{i=1}^{n} q_i} \tag{15}$$

where C_i and q_i designate the equilibrium concentration and uptake of component i, respectively.

Figure 4 compares the prediction data of Cu^{2+}–Ni^{2+} system with the experimental data. It is seen that the IAST and the Sips models result in close prediction of the isotherm of this system as compared to experimental data.

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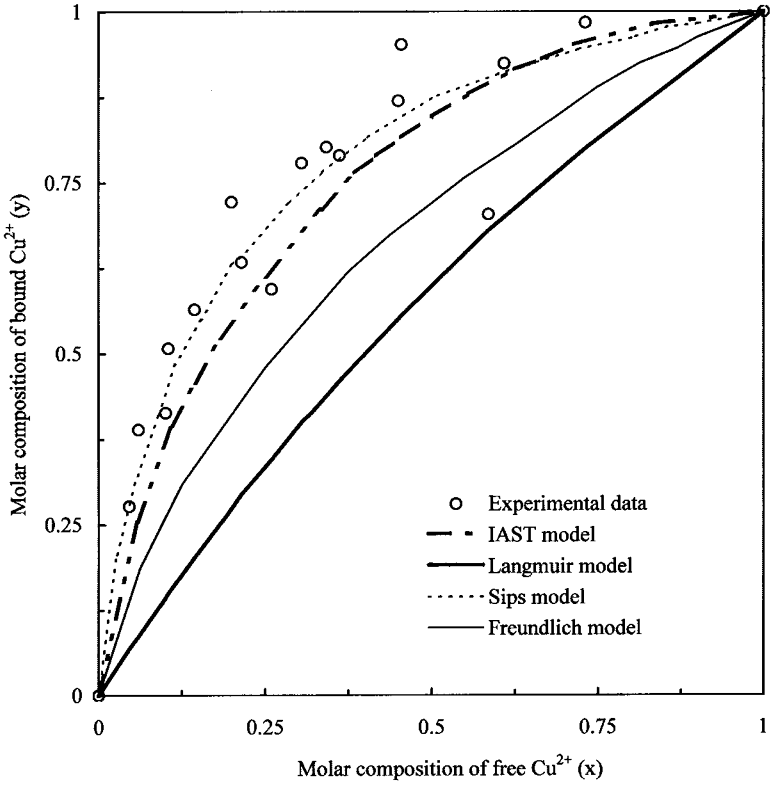

Figure 4. Prediction of binary equilibrium data of Cu^{2+}–Ni^{2+} system using different type of multicomponent isotherm models.

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However, Langmuir and Freundlich type models did not closely follow the experimental data.

For the binary system Cu^{2+}–Zn^{2+}, generally all of the models except Langmuir showed reasonable prediction of the equilibrium data as compared to the experimental data (Fig. 5). Although this model (Langmuir) successfully fitted the single isotherm data of both Cu^{2+} and Zn^{2+}, it failed in the prediction of their binary sorption isotherm.

In Fig. 6, it is obvious that the Sips, IAST, and Freundlich models are the convenient models to predict the isotherm of Ni^{2+}–Zn^{2+} binary system. The results of Fig. 6 show that the Langmuir model is very poor in predicting the Ni^{2+}–Zn^{2+} experimental data. Although this model successfully fitted the single isotherm data of both Ni^{2+} and Zn^{2+}, it failed in predicting their binary sorption

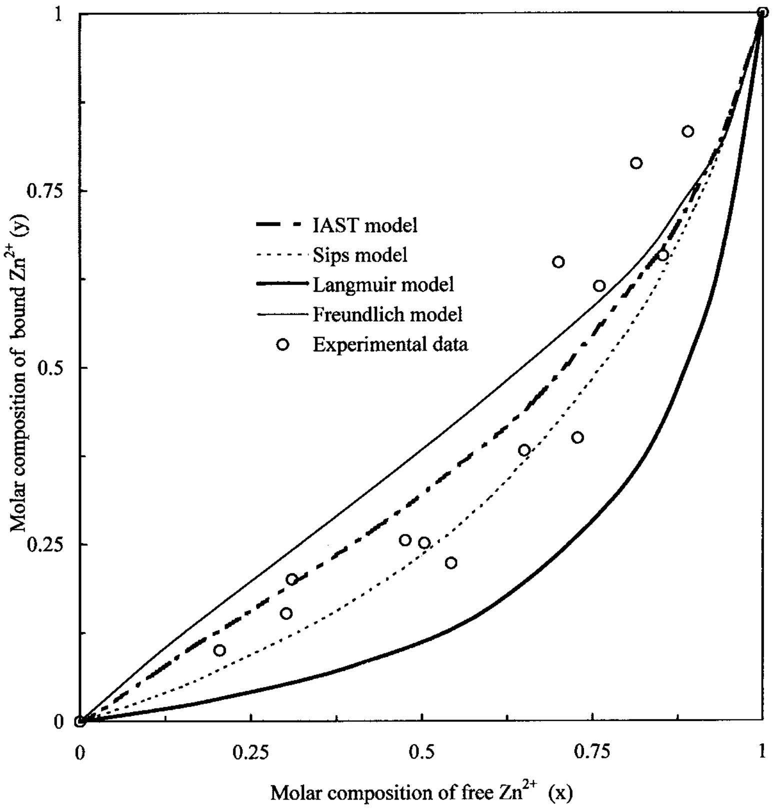

Figure 5. Prediction of binary equilibrium data of Zn^{2+}–Cu^{2+} system using different type of multicomponent isotherm models.

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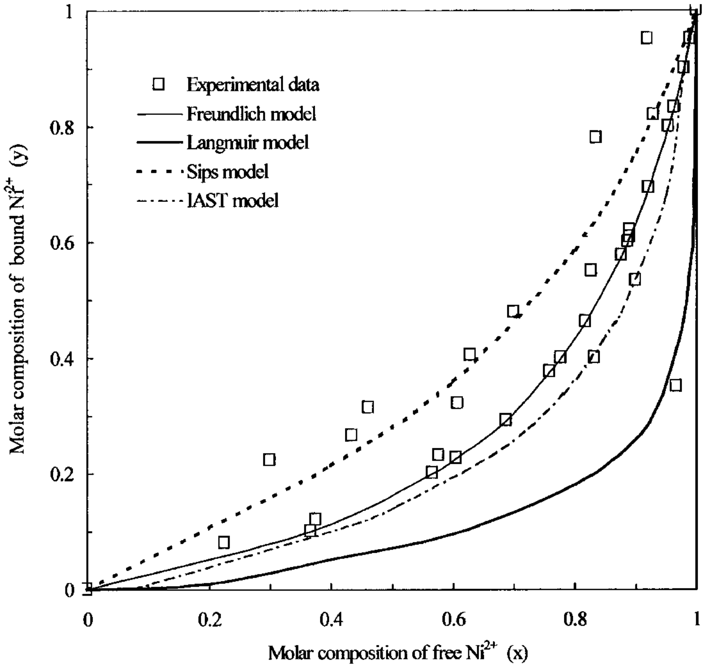

Figure 6. Prediction of binary equilibrium data of Ni^{2+}–Zn^{2+} system using different type of multicomponent isotherm models.

isotherm. However, the predictions by all of the models were poor at low metal concentrations. This is a characteristic of adsorption isotherm models, where the behavior of most of the models is linear at very low concentrations. The Freundlich isotherm model was closer to the experimental data than Langmuir isotherm, most likely because of its specific trend and applicability. The different k_L values of Ni^{2+} and Zn^{2+} could be the reason for the deviation of the extended Langmuir model prediction from the experimental data.

The effect of the secondary metal on the uptake of the primary is displayed in Fig. 7. For the following discussion, two initial concentrations of the primary metal ions have been chosen to represent "low" (40 ppm) and "high" (60 ppm) initial concentrations. Figure 7A shows that at low or high initial concentrations of Cu^{2+}, its uptake was reduced by 8 and 4%, respectively, when the initial concentration of Ni^{2+} was 10 ppm. However, this reduction was increased to 30 and 22% when the initial Ni^{2+} concentration reached a value of 60 ppm. Similar trends can be seen for the adsorption of zinc and nickel at different initial concentrations of cocations (Fig. 7B and C).

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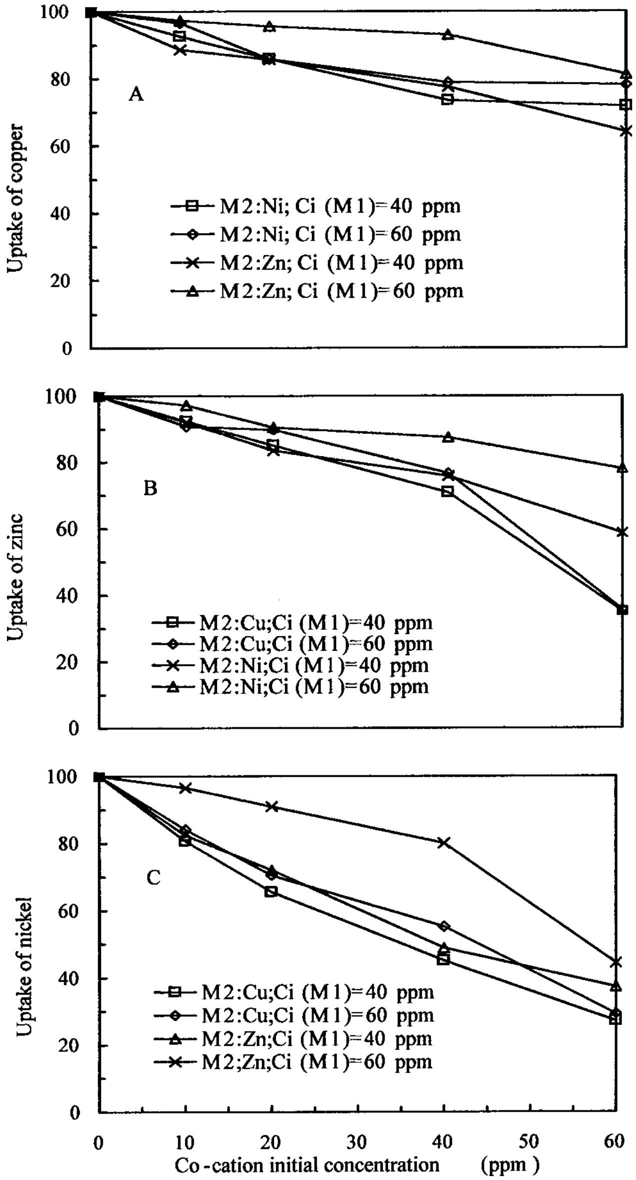

Figure 7. Effect of cocation (M2) initial concentration on the uptake of the primary cation (M1) at its initial concentration (M1).

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Ternary-Metal Sorption

The sorption of metal ions from the ternary system Cu^{2+}–Zn^{2+}–Ni^{2+} was also considered in this work. The effect of ionic interaction on the sorption process can be represented by the ratio of the sorption capacity for one metal ion in the ternary system, q_T, to the sorption capacity for the same metal in the binary system, q_B (17). The sorption capacities of the metal ion in the ternary, A–B–C, and binary, A–B, systems were determined by measuring the concentration of the metal ion, A, in the residue of the ternary and binary mixtures, respectively, after attaining equilibrium.

If

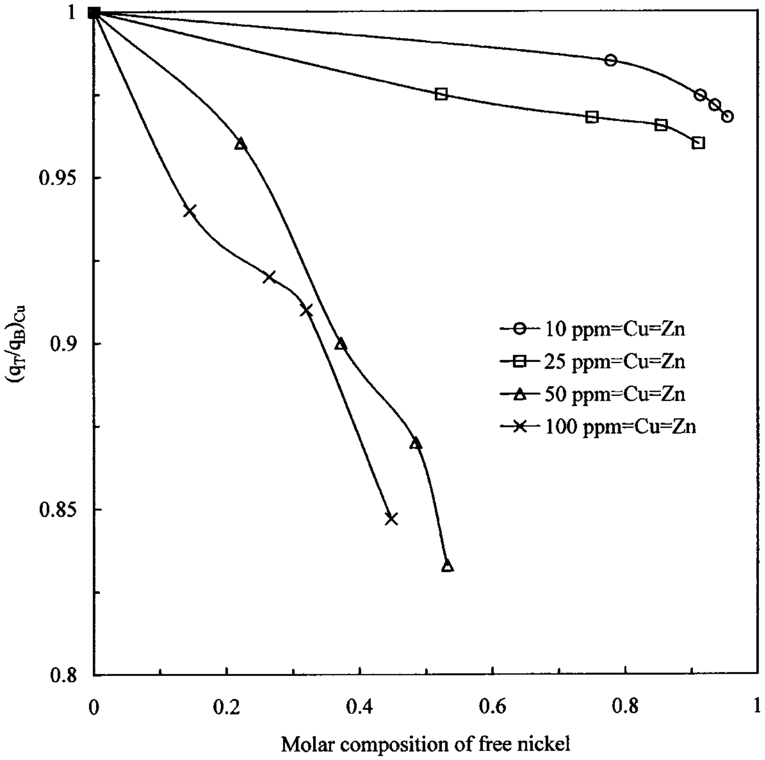

Figure 8. Effect of Ni^{2+} concentration on the uptake of Cu^{2+} in the ternary system Cu^{2+}–Zn^{2+}–Ni^{2+} using different initial concentrations of Cu^{2+} and Zn^{2+}. q_T/q_B represent the ratio of Cu^{2+} uptakes in the ternary system Cu^{2+}–Zn^{2+}–Ni^{2+} to that in the binary system Cu^{2+}–Zn^{2+}.

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$(q_T/q_B) > 1$ sorption is promoted by the presence of other metal ions,
$(q_T/q_B) = 1$ no observable net interaction effect,
$(q_T/q_B) < 1$ sorption is suppressed by the presence of other metal ions.

It was noticed that the ratio of copper uptake in the ternary system Cu^{2+}–Zn^{2+}–Ni^{2+} to that in the binary system Cu^{2+}–Zn^{2+} decreases as the equilibrium molar composition of nickel in solution increases at fixed initial concentration of Cu^{2+} and Zn^{2+} in the solution (Fig. 8). In addition, it is noticed that this ratio decreases more appreciably when the initial concentrations of Cu^{2+} and Zn^{2+} in the solution increase. Therefore, the sorption is suppressed by the presence of other metal ion.

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CONCLUSIONS

The results show that bones can be used for sorption of the following metals in the indicated order (molar basis) in the single systems: $Cu^{2+} > Zn^{2+} > Ni^{2+}$. In the binary and ternary systems, the sorption is suppressed by the presence of other metal ions in the aqueous solution.

Generally speaking, the prediction of multicomponent adsorption isotherm models of Cu^{2+}–Ni^{2+} binary system is relatively close to the experimental data. In the Cu^{2+}–Zn^{2+} binary system, generally all of the models except Langmuir showed relatively good prediction of the equilibrium data as compared to the experimental data. Although this model (Langmuir) successfully fitted the single isotherm data of both Cu^{2+} and Zn^{2+}, it failed in the prediction of their binary sorption isotherm. For the Ni^{2+}–Zn^{2+} binary system, the Sips, IAST, and Freundlich models are convenient to predict the isotherm, whereas the Langmuir is very poor in predicting the Ni^{2+}–Zn^{2+} experimental data. Although these models successfully fitted the single isotherm data of both Ni^{2+} and Zn^{2+}, they failed in prediction of their binary isotherm.

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