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# Prediction of the Dynamic Adsorption Behaviors of the Uranium and Cobalt Ions in a Fixed Bed by Surface Modified Activated Carbon

# 표면개질 활성탄을 이용한 고정층에서 우라늄 및 코발트 이온의 동적 흡착거동 모사

**Geun-IL Park*, Jung-Won Lee, Kee-Chan Song, In-Tae Kim, Kwang-Wook Kim, and Myung-Seung Yang**
Korea Atomic Energy Research Institute, 150 Duck-Jin Dong, Yusung-Ku, Daejeon
ngipark@kaeri.re.kr

박근일, 이정원, 송기찬, 김인태, 김광욱, 양명승
한국원자력연구소, 대전광역시 유성구 덕진동 150번지

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## Abstract

In order to predict the dynamic behaviors of uranium and cobalt in a fixed bed at various influent pH values of liquid waste, the adsorption system is regarded as a multi-component adsorption between each ionic species in the solution. Langmuir isotherm parameters of each species were extracted by incorporating equilibrium data with the solution chemistry of the uranium and cobalt using IAST. Prediction results were in good agreement with the experimental data, except for a high concentration and pH. Although there was some limitations in predicting the cobalt adsorption, this method may be useful in analyzing a complex adsorption system where various kinds of ionic species exist in a solution.

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**Key words** : Surface-modified activated carbon, Uranium, Cobalt, Adsorption, Dynamic behavior

## 요 약

폐기물 용액의 pH 변화에 따른 고정층에서 우라늄 및 코발트 이온의 흡착거동을 다성분 흡착 시스템으로 가정하여 이론적으로 예측하였다. 즉 pH 변화에 따라 존재 분율이 달라지는 각 이온 성분들이 상호 경쟁적으로 흡착한다는 가정하에서, 평형실험에서 얻어진 결과와 우라늄 및 코발트 이온의 용액특성 (Solution chemistry)을 상호 결합하여 각 이온 성분들의 Langmuir 평형상수 값을 Ideal Adsorbed Solution Theory를 도입하여 구하였으며, 이상의 결과를 이용하여 고정층 파과곡

선을 이론적으로 계산한 결과 pH 변화에 따른 흡착거동을 비교적 잘 예측할 수 있었다. 따라서 본 연구에서 시도한 방법은 이온 농도와 pH가 높은 경우를 제외하고 pH 변화에 따라 용액 내에 이온의 형태가 다양하게 존재하는 흡착 시스템을 이론적으로 예측하는 데 비교적 유용하게 사용할 수 있을 것으로 판단된다.

**Key words** : 표면개질 활성탄, 우라늄, 코발트, 흡착, 동적거동

## Ⅰ. Introduction

Various kinds of radioactive fission or corrosion products as well as uranium are simultaneously present in liquid wastes, and exist in several characteristic forms[1,2]. These are generated during the fabrication and reprocessing of nuclear fuels, decommissioning of alpha contaminated facilities, and other activities[3]. In particular, because of its long half-life and high radiological toxicity, uranium is considered to be a serious long-term environmental hazard. Cobalt is considered to be one of the key radionuclides contributing to the radiation dose for workers and the public[4]. Like uranium, a very low concentration of cobalt is allowed in the effluents from a nuclear facility[5]. In order to avoid the problems arising from the contamination of radioactive nuclides, both ion exchangers and sorbents for improving the removal efficiencies have been used. Numerous experimental studies have also been conducted on the removal of residual uranium and cobalt from radioactive liquid wastes [6-8].

Commonly available methods for the removal of uranium and cobalt from aqueous solutions includes sorption using synthetic polymer resin, activated carbon, or biosorbent. The acid cation exchange resin offered prospects for an extended useful life for the treatment of liquid wastes[1]. However, it suffers from degradation or enhanced polymerization of the resin, depending on the level of radioactivity during the operation. Activated carbon, on the other hand, is about one-tenth the cost of the resin on an equivalent volume basis, and it would not have the properties for degradation or polymerization. Therefore, activated carbon as an inorganic adsorbent has often been used to remove the radionuclides from the liquid effluents, due to its advantages of a high chemical, radiological and thermal stability over polymeric materials[9]. Activated carbon has a strong affinity for organic molecules and contains oxygenated functional groups on its entire surface, which are capable of sorbing metal ions from an aqueous solution[10,11]. Despite this, little work has been done in determining the use of activated carbon in the sorption of radionuclides from aqueous solutions. This comes from the significant reason that activated carbon can adsorb only a small quantity of radionuclides in an acidic solution, as compared with an ion exchange resin[1]. From this point of view, many attempts have been made to enhance the adosrption capacity of activated carbon[12,13]. Numerous studies have been conducted to connect the surface characteristics of oxidized carbons or fibers with an influence on the adsorption capacities of the toxic gases and organic or metal pollutants in the waste on these sorbents[11,14]. It has been reported that the effect of oxidation treatment of a carbon surface has often manifested the removal of a heavy metal ion from an aqueous solution[6,8,15]. These results may lead to the conclusion that both the pore structure and surface chemistry of carbon affects significantly the adsorption characteristics of inorganic and organic polar molecules. The nature and the capacity of surface oxides formed on the treatment with oxygen depend on the nature of the carbon and the history of its formation. In case of oxidative treatments in solutions, the major reaction is the formation of the surface oxygen compounds. According to many workers[5-7], the adsorption sites of metal ions on activated carbon are known as oxide groups such as carboxyl and phenolic groups and increased by surface acid treatment. The formation of these surface compounds using various types of carbon and using different oxidative treatments in gaseous and liquid phases has been studied by a large number of investigators and has been well reviewed[5-7]. Park et.al have experimentally studied the adsorption characteristics of uranium and cobalt ions in an acid solution on oxidized activated carbon[8].

The prediction of the dynamic behaviors in practical column process generally depends on the incorporation of the adsorption equilibrium relationship and the transport mechanism with a mass balance. In the adsorption of metal ions from an aqueous solutions, solution pH may significantly affect the extent of adsorption[15]. Therefore, surface complexation models are useful to quantify the metal ion adsorption on a hydrous solid surface[16]. While the use of the complexation hypothesis in correlating the metal ion adsorption data has been found to be useful, this approach also has its deficiencies. On considering the prediction of the adsorption behaviors in a continuous-flow column operation, an equilibrium model to calculate the adsorption amount of a specific ion over a wide range of variables should be combined with a transport model in a column operation. In this case, the large number of parameters, presented in the surface complexation models, often makes it difficult to obtain the stability of a numerical solution, and to ascertain the true physical significance of these parameters.

In this paper, a theoretical approach for analyzing the effect of pH on the dynamic behaviors in a fixed bed packed with surface-modified activated carbon was performed using a generalized adsorption model. Isotherm parameters of each uranium species were estimated by the combination of the equilibrium data with the solution chemistry using a minimizing routine. The prediction of the adsorption breakthrough curves with the variation of the influent solution pH in a fixed bed packed with surface modified activated carbons was performed.

## Ⅱ. Experimental

The granules of activated carbon, obtained from Han-il Green Tech. Co., were ground, and sieved to give an average particle size of 0.85mm from the sieve openings. Some of the pre-treated activated carbons were oxidized by heating in a 7 N nitric acid solution at temperature of 80~90 ℃ for 10 hours in a volumetric flask[6,8]. Fixed bed runs were confined to the surface-modified activated carbons. The adsorption experiments in a fixed bed were carried out using a glass column of 0.01m inside diameter and 0.3 m in length. Pretreated adsorbents were packed into the column and sustained by glass beads. A micro-metering pump was used to maintain the constant down-flow of 2 ml/min with a range of influent solution pH(pHi) from 3 to 6. Effluent from the column was collected periodically by a fraction collector, and the pH of the collected samples was directly measured, except for the over-night runs.

## Ⅲ. Theoretical Approach

In the adsorption of metal ions from an aqueous solutions, the solution pH may significantly affect the extent of adsorption. The total adsorption includes both the ionized as well as the nonionized species of the adsorbate, and the electrostatic field between the solution and the charged adsorbent surfaces may also play a role in determining the adsorption equilibrium. Generalized isotherm equations such as the Langmuir or Freundlich equations can be used to predict the equilibrium adsorption amounts in a specific system. However, these isotherm equations obtained from a data-fitting cannot be used to predict the effect of various solution variables such as the pH, ionic strength, and type of electrolyte, nor the effect of electrostatics on the extent of adsorption. The general approach in predicting the equilibrium adsorption amount of a metal ion in an aqueous solution is the adsorption models based on the chemical reaction or the complexation models[15-17]. Surface complexation models have been developed to accurately quantify the metal ion adsorption on adsorbent surfaces. While various model parameters used in this model can only be obtained by fitting experimental data, it is nevertheless useful because it can simulate the adsorption behavior over a wide range of variables. Most of the works related to the prediction of metal ion adsorption by surface complexation models have been confined to the analysis of equilibrium data. While the use of the complexation hypothesis in the correlating metal ion adsorption data has been found useful, this approach also has its deficiencies. On considering the prediction of the adsorption behaviors in a continuous-flow column operation, an equilibrium model to calculate the adsorption amount of a specific ion over a wide range of variables should be combined with the transport model in the column. In this case, the large number of parameters, presented in the surface complexation models, often makes it difficult to obtain the stability of a numerical solution, and to ascertain the true physical significance of these parameters. Moreover, if the adsorption system has a high adsorption affinity to the metal ion, this difficulty becomes serious due to the system's stiffness and causes a very long computing time.

### 1. Extraction of Isotherm Parameters for Each Species

On considering of the available models for the surface charging, the electrical double layer and the possible complexation model for the ions, it is obvious that for a given metal ion-adsorption system, there exists, at least theoretically, several possibilities for describing the adsorption equilibrium and correlating the isotherm data. The use of an excessively large number of parameters can be rule out for a practical consideration. With the help of the generalized Langmuir equation, some possibilities to represent the equilibrium data involving the pH effect on the metal adsorption exist. The Langmuir isotherm is based on the simplest one of several theories that are available to describe the relationship between the amount of a solute adsorbed onto a surface($q$) and the concentration of the solute in the liquid phase($C$)[17].

$$\Theta = \frac{q}{q_m} = \frac{BC}{1+BC} \qquad (1)$$

where $q_m$ is a temperature-independent constant which is supposed to represent a fixed number of surface sites and B is a temperature dependent Langmuir equilibrium constant.

For the convenience of a simple calculation, it is assumed that the competitive adsorption of the three components are as follows ; $UO_2^{2+}$. and $UO_2(OH)^+$ as a monomer, $(UO_2)_2(OH)_2^{2+}$ as a dimer, and $(UO_2)_3(OH)_5^+$ as a trimer at around pH 6 or below, as shown in Fig.1. This computation was performed using MINTEQ2 code that was developed to apply fundamental principles of thermodynamics to solve chemical equilibria [18]. MINTEQ2 code can calculate ion specification and solubility, adsorption and precipitation/dissolution of solid phases at equilibria. Therefore, the total adsorption amount of uranium is the sum of each component adsorption amount. On considering the above assumptions, the uranium adsorption system in an aqueous solution would be regarded as the multi-component adsorption of each uranyl hydroxyl species. Once we obtain the equilibrium pH and the total adsorption amount from an equilibrium experiment over a wide range of pH, the equilibrium parameters of each uranyl hydroxyl species can be calculated by the combination of the solution chemistry of the uranium with the pH variation with IAST(Ideal Adsorption Solution Theory)[17,19]. Models or correlations for multicomponent adsorption are crucial to the design of adsorptive component separation processes. They should be capable of predicting the adsorbed amount at equilibrium from single component isotherms for each constituent in the mixture, within a given range of operating concentration. Among the theories, the thermodynamic methods are independent of the specific physical model of adsorption. The thermodynamic analysis can predict multicomponent equilibrium properties from pure component isotherms. All of the thermodynamic methods to predict the multicomponent adsorption equilibria assume ideal adsorbate behavior. Based on the Raoult's law which describes vaporliquid equilibrium, IAST describes vaporsolid equilibrium in a typical case of adsorption.

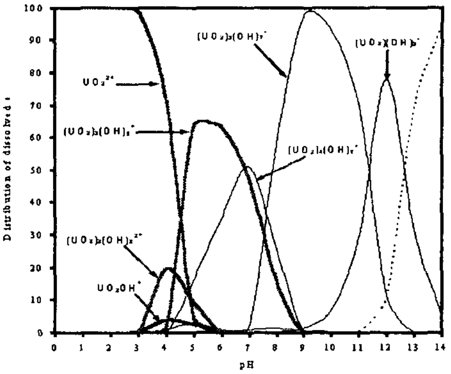

**Fig.1.** Distribution of the uranyl-hydroxyl complexes vs pH in pure water at 30℃ and the total uranium concentration of $10^{-3}$ mol/l.

The Langmuir equation is adopted for calculating the equilibrium adsorption amount of each uranyl hydroxyl species with a variation of the pH in a range from 3 to 7. A mutual competitive adsorption between each species is represented by IAST. For the uranium adsorption system, the isotherm parameters for each ionic species (monomer, dimer and trimer) are listed in Table 1. For cobalt, the isotherm parameters of each species are confined to the adsorption system on activated carbon, as listed in Table 1.

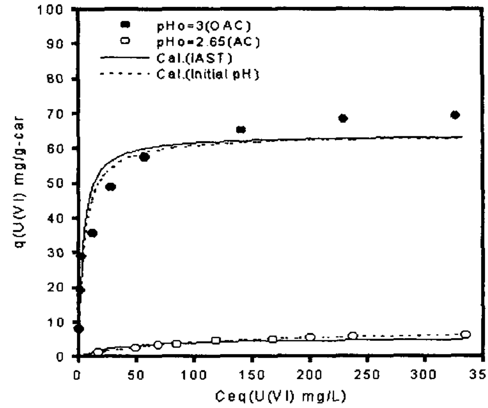

**Fig. 2.** Effect of the isotherm parameters on the prediction of the experimental equilibrium data obtained at a low pH.

Fig. 2 represents the comparison of two results in the low pH ranges. The solid line indicates the result predicted by IAST, the dashed line represents the one by a simple Langmuir equation expressed in terms of the total concentration.

A dynamic model for the uranium adsorption in fixed beds packed with oxidized and activated carbon was formulated. The generalized equations consist of the transport equation and the mass balance equation for the adsorbed component in a multi-component system.

**Table 1. Langmuir isotherm parameters of each uranyl species and cobalt ions calculated by IAST**

| System<br>Species | As-received activated carbon | | Oxidized carbon | |
|---|---|---|---|---|
| | $q_m$(mol/m$^3$) | B(m$^3$/mol) | $q_m$(mol/m$^3$) | B(m$^3$/mol) |
| U(monomer) | $2.2\times10^{-2}$ | 7.42 | 0.268 | 65.2 |
| U(Dimer) | $5.1\times10^{-2}$ | 23.8 | 0.35 | 50.2 |
| U(Trimer) | $1.14\times10^{-1}$ | 103 | 0.67 | 30.0 |
| $Co^{2+}$ | $4.8\times10^{-2}$ | 1.06 | - | - |
| $Co(OH)^{+}$ | $9.4\times10^{-2}$ | $1.1\times10^{5}$ | - | - |

## 2. Dynamics of Fixed Bed Adsorption

Mass balance for the mobile phase

The transport equation and the relevant initial and boundary conditions for a multi-component of the mobile phase in the column can be written as follows.

$$\frac{\partial C_{bi}}{\partial t} = D_L \frac{\partial^2 C_i}{\partial x^2} - v \frac{\partial C_i}{\partial z} - \frac{1-\varepsilon_b}{\varepsilon_b} \frac{3k_f}{R}(C_{bi} - C_{si}) \qquad (2)$$

$$C_{bi} = C_{bi}(0, z) \qquad (3)$$

$$D_L \frac{\partial C_{bi}}{\partial t} \mid_{z=0} = -v(C_{bi} \mid_{z=0} - C_{fi}(t) \mid_{z=0}) \qquad (4)$$

Mass balance for the solid phase

$$\frac{\partial q_i}{\partial t} = D_{si}\left[\frac{\partial^2 q_i}{\partial r^2} + \frac{2}{r}\frac{\partial q_i}{\partial r}\right] \qquad (5)$$

$$q_i = q_i(0, r) \qquad (6)$$

$$\frac{\partial q_i}{\partial r} \mid_{r=0} = 0 \qquad (7)$$

$$\frac{\partial q_i}{\partial t} = 0 \qquad (8)$$

Nomenclatures mentioned above equations are expressed as following.

$C_{bi}, C_{fi}$ : concentration of ith adsorbate in bulk solution or fluid phase in a multi-component system, mg/ℓ or mol/m$^3$.

$C_{si}$, : saturation concentration of an ith adsorbate in the liquid phase in a multi-component system, mg/ℓ or mol/m$^3$.

$\upsilon$, $D_L$, $k_f$, $\varepsilon_b$ : interstitial velocity of the fluid in a fixed bed(m/s), axial dispersion coefficient in a fixed bed(m$^2$/s), external film mass transfer coefficient(m/s), void fraction in fixed bed.

$q_i$ : equilibrium amount adsorbed of ith component; Amount of adsorbates adsorbed in equilibrium with initial concentration, mg/g or mol/kg. t, z, r : time, axial distance in a fixed bed, distance in a solid particle.

To simplify the equations discussed above, a set of equations can be written in a dimensionless form by introducing dimensionless groups and variables. The estimated value of the axial dispersion coefficient (DL) is 3.42 x $10^{-7}$ m$^2$/s for uranium-carbon system, and 3.45 x $10^{-7}$ m$^2$/s for cobalt-carbon system. Also, the external film mass transfer coefficient, kf, estimated from Eq. (2) is 8.14 x $10^{-6}$ m/sec for uranium, 1.14 x $10^{-5}$ m/sec for cobalt.

The techniques of an orthogonal collocation for the finite elements combine the classical procedure of an orthogonal collocation with the high accuracy of the finite element method. Owing to the system stiffness, its integration is made by means of a stiff solver : LSODI of the International Mathematics and Science Library (IMSL).

## Ⅳ. Results and Discussion

In order to demonstrate the applicability of the prediction method adopted in this study, some breakthrough curves of the uranium and cobalt adsorption on activated and oxidized carbons under different conditions are predicted. Main operating parameters were the input concentrations adjusted to different pH values, and fixing other operating variables such as the flow rates and bed depths.

The breakthrough curves of uranium ions at the influent pHi of 3~5 using the oxidized carbon and activated carbon were obtained at uranium concentrations of 50 and 200 mg/*l*. Figs. 3 and 4 show a comparison of the experimental data with the prediction results when the input concentration was 0.21 mol/*m*$^3$ (50 mg U(VI)/*l*). The performance of activated carbon for uranium removal in a fixed bed was very low compared with that of oxidized carbon. At the influent pH 3, the breakthrough curve showed the common S-shape which is typical for a single species adsorption on activated carbon. However, when the influent pHi becomes higher, there are unusual breakthrough behaviors, such as the trailing and plateau regions, which are normally represented by a multicomponent system with a competitive adsorption[13,17]. In the case of oxidized carbon, the effluent pH decreased in the earlier adsorption stage, and then increased to the influent pHi as the adsorption proceeded, as typically represented in Fig. 5. Needless to say, even though the effluent pH does not stand for the pH variation inside the column, it can be inferred as overall characteristic of the column's inside. At pH 5 and a uranium concentration of 200 mg/*l*, the effluent pH sharply decreased to about 3 and then rapidly increased, when compared with the pH variations at the concentration of 50 mg/*l*. It was observed that the breakthrough time in each run might have some relationship with the reflection point of the pH change as the adsorption proceeds. As discussed previously, the rapid decrease of the effluent pH in the earlier adsorption stage also implies that great amounts of uranium were removed by the ion-exchange mechanism with the hydronium ions of the surface functional group[10]. It is thought that the trailing behaviors in the breakthrough profiles are closely related to the difference in the adsorption capacity with the pH variation of the uranium solution. The rapid decrease of the pH in the earlier adsorption stage leads to a decrease of the adsorption amount of the uranium, mainly due to the pH effect. As the adsorption proceeds, the solution pH in a fixed bed increases and the adsorption amount becomes higher. On the contrary, at pH 3 of the influent solution, the pH variation in the column packed with activated carbon showed a rapid increase up to pH 6.5 in the earlier adsorption stage and a sharp decrease as the adsorption proceeded. Therefore, in order to predict the adsorption characteristics of uranium in a fixed bed, the pH influence should be considered. However, it is difficult to consider the pH variation with the distance of fixed bed for the prediction of breakthrough curve. Therefore, the linear relationship between the constant influent solution pH and the pH value of effluent solution obtained from experiment with the adsorption time was assumed in this paper. In case of uranium adsorption on activated carbon, the main uranium species involved in adsorption would consider to be consisted of four types as mentioned before. For the convenience of simple calculation, it is assumed that the concentration of $UO_2(OH)^+$ compound is including in $UO_2^{2+}$. That is, competitive adsorption of three components are considered as follows ; $UO_2^{2+}$ and $UO_2(OH)^+$ as monomer, $(UO_2)_2(OH)_2^{2+}$ as dimer, and $(UO_2)_3(OH)_5^+$ as trimer at around pH 6 or below. Therefore, total adsorption amount of uranium is the sum of each component adsorption amount. This assumption represents no interaction of each species during adsorption. Once we know the equilibrium pH and total concentration of uranium, the concentration of each uranyl hydroxyl species can be calculated from the information of solution chemistry of uranium with pH variation using the hydrolysis reaction constants (pKi values). On considering above assumptions, uranium adsorption system in aqueous solution would be regarded as the multicomponent adsorption of each uranyl hydroxyl species. The concentration of individual species would be changed with equilibrium pH.

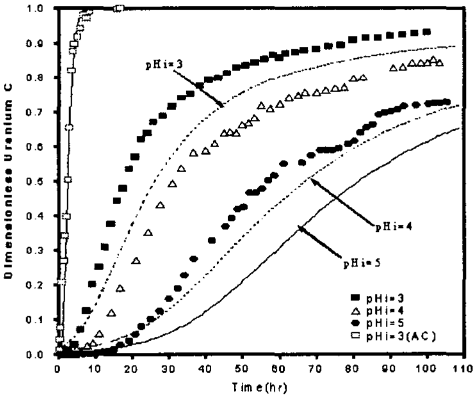

Fig.3. Comparison of the experimental data of the uranium adsorption on oxidized carbon in a fixed bed with the prediction results obtained without introducing the competitive adsorption of each species.[Experimental conditions : $Co=0.21\ mol/m^3$, pHi=3, 4, 5]

Prediction in Fig. 3 was calculated by the isotherm equation obtained as a function of the total uranium concentration, without considering the competitive adsorption of each ionic species. Large discrepancies between the prediction and experimental data are observed, with the exception of the breakthrough curve at pHi 3. These results may come from the inaccurate calculation of the adsorption amount with the variation of the pH. The success of the theoretical prediction, in general, depends on how to incorporate the adsorption equilibrium relationship and the transport mechanism. As discussed earlier it is difficult to have an equilibrium data set equivalent to the influent pH from an equilibrium experiment. Therefore, a rigorous calculation leads to a poor agreement with the experimental data. Fig. 4 represents a comparison of the experimental data with the model prediction, which was obtained by introducing the competitive adsorption of each uranyl hydroxyl species based on the isotherm parameters in Table 1. Agreement between the experimental and prediction curves was fairly good.

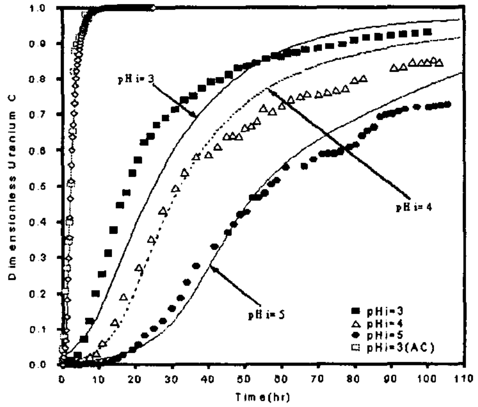

**Fig. 4.** Comparison of the experimental data of the uranium adsorption on oxidized carbon in a fixed bed with the prediction results obtained from the competitive adsorption of each species.[Experimental conditions: Co=0.21 mol/$m^3$, pHi=3, 4, 5]

For the cobalt adsorption system, the dynamic behavior of cobalt on activated carbon was satisfactorily predicted by considering the competitive adsorption of each species, as shown in Figs. 6 and 7. However, because the isotherm parameters of each ionic species in the oxidized carbon-cobalt adsorption system could not be obtained as explained earlier, the method adopted in this study has some limitations in predicting the dynamic behaviors in a simple system.

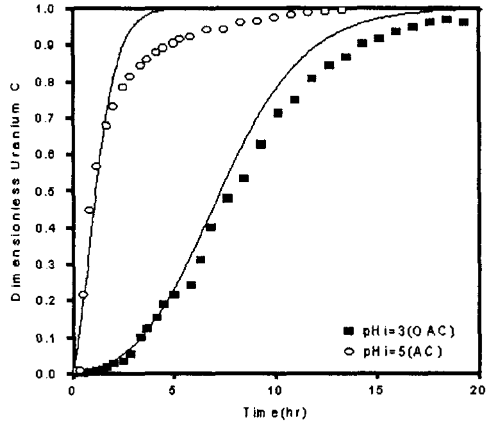

**Fig. 6.** Comparison of the experimental with the prediction results for the cobalt adsorption on carbons in a fixed bed[Experimental conditions : Co=0.21 mol/$m^3$, pHi=3, 5]

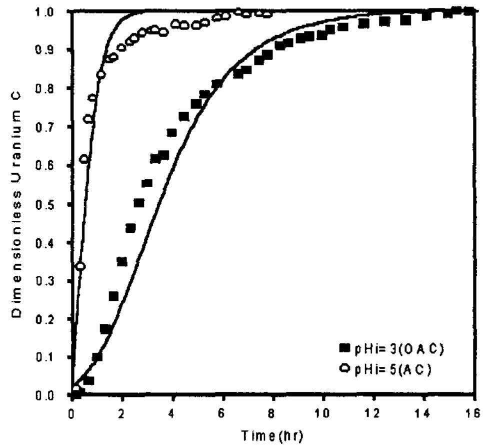

**Fig. 7.** Comparison of the experimental with the prediction results for the cobalt adsorption on carbons in a fixed bed[Experimental conditions: Co=0.84 mol/$m^3$, pHi=3, 5]

Even if only a single ion exists over the specified pH ranges, the adsorption capacity of cobalt is increased due to the increase of the deprotonation onto the carbon surface as the pH becomes higher. However, at the low pH ranges, the prediction result gives a good fit to the experimental data. It was observed that the equilibrium pH was nearly constant over a wide range of equilibrium concentrations when the initial pH was fixed at 3. Therefore, this isotherm equation may be used in the prediction of dynamic behavior at a pH 3 of influent solution. Fig. 8 shows the experimental and predicted breakthrough curves in binary adsorption system under the input concentration of the uranium of 0.84 mol/$m^3$ and the cobalt of 0.21 mol/$m^3$ at a pH 3 of the influent solution. The excess curve of cobalt ion implies low affinity with oxidized carbon surface at same molar concentration in binary-component system, and uranium exhibited no excess concentration. This result provides uranium has high affinity with oxidized carbon. In binary system, the weakly adsorbed component competes with and is replaced by the strongly adsorbed component. However, displacement effect was reduced when the molar ratio between the weakly adsorbed and strongly adsorbed component was low or high. It was also observed that the estimated kinetic parameters in a fixed bed was a little higher than that in the batch adsorber.

As a whole, the prediction method adopted in this study for predicting the dynamic behavior incorporated with the pH influence may be applied to a complex system where various kinds of species exist in a solution.

## V. Conclusions

While a surface complexation model is useful for predicting the adsorption behaviors of a metal ion, the use of an excessively large number of parameters can rule it out for a practical consideration, especially a column operation. Therefore, for describing the adsorption equilibrium and correlating the isotherm data, an alternative approach for representing the equilibrium data considering the pH effect on a metal adsorption was suggested using a generalized Langmuir equation. In the uranium adsorption system, the competitive adsorption of three kinds of uranyl hydroxyl species was considered. Based on the results from the equilibrium studies, the equilibrium isotherm parameters of each uranyl hydroxyl species were extracted by the combination of the solution chemistry of the ranium with the pH variation with IAST. Experimental breakthrough curves at various influent pH's was satisfactorily predicted by introducing the competitive adsorption of each uranyl hydroxyl species, with the exception of the breakthrough curve at pHi 5. Although some limitations in predicting the cobalt adsorption system was observed, this method may be useful in analyzing a complex

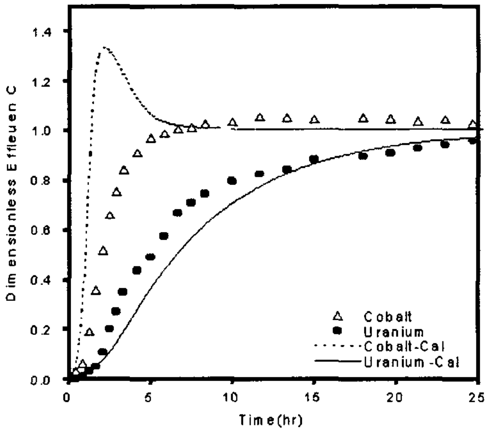

**Fig. 8. Prediction of the breakthrough curves for binary adsorption on oxidized carbon at input concentration of uranium(0.84 mol/$m^3$) and cobalt(0.21 mol/$m^3$), pHi=3.**

adsorption system where various kinds of ionic species exist in a solution.

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