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Kinetic Model for the Decomposition of Cyanide during the Elution of Gold from Activated Carbon

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ABSTRACT

Free cyanide is usually present during the elution of gold cyanide from activated carbon. The decomposition of cyanide is important not only in the extraction of gold but also for environmental reasons. Previous studies have indicated that competitive adsorption of cyanide with aurocyanide plays a minor role at the elevated temperatures used in industry. A more important effect of cyanide is its decomposition reaction with functional groups on the carbon, the products of which passivate the surface for adsorption of aurocyanide, and thereby cyanide appears to enhance the elution of aurocyanide. It was observed that the degree of passivation affects the elution of gold and the degradation/adsorption of cyanide itself. Batch tests have shown that the rate of oxidation of cyanide at low temperatures is independent of particle size and more dependent on film transfer. Based on these observations, a kinetic model was proposed for the decomposition and elution of cyanide in packed columns of activated carbon. The oxidation and hydrolysis reactions in the bulk solution, as well as in the carbon pores, were combined and described by single first-order reactions. It was found that the same kinetic parameters could be used to give satisfactory predictions of experimental data for a wide range of conditions.

INTRODUCTION

Cyanide is used as a case-hardening agent in metallurgy and is also used widely in the electroplating industry. In view of the mandatory removal of cyanides from the effluents of these industries, numerous methods for the oxidative destruction of cyanide, including electrochemical methods (1, 2), have been devised. Cyanide is also the main lixiviant used world-

wide for the extraction of gold from its ores. Activated carbon is utilized for the adsorption of gold cyanide from the leached slurries. It is well known that the free cyanide also adsorbs onto the activated carbon and decomposes via oxidation on the surface of the carbon and/or hydrolysis in the bulk solution. Gold cyanide is normally eluted from the loaded carbon by pretreatment in caustic cyanide solutions at elevated temperatures. Previous studies (3–5) have indicated that the decomposition products of cyanide passivate the surface of the activated carbon and thereby enhance the desorption of gold cyanide. Therefore, knowledge about the mechanism and kinetics of the decomposition of cyanide is essential, not only from an environmental perspective but also for the optimization of separation processes such as the elution of gold from activated carbon.

There are two main methods of elution used in the gold industry: 1) The Zadra process in which hot cyanide solution is circulated through a stripping column and electrowinning cell (a US Bureau of Mines invention); and 2) The Anglo American Research Laboratory (AARL) method in which loaded carbon is pretreated in a column with hot caustic cyanide solution followed by elution with hot deionized water (a South African invention). Cyanide and hydroxide salts are added during both the AARL and Zadra elution procedures to enhance the elution of the gold cyanide. The extent to which cyanide is required in the elution step is determined by the degree of irreversibility of the adsorbed gold due to acid treatment prior to elution (6, 7). The equilibrium between the adsorbed gold and the gold cyanide in solution is affected by the concentrations of these additives, which change continuously during a run (3, 4). Both the equilibrium of adsorption/desorption as well as the degradation of the cyanide are affected significantly by temperature (8). Hence, it is imperative that the elution profile of cyanide is known in order to simulate the profile of gold elution (4).

A number of papers have appeared on the mechanism and kinetics of the decomposition of cyanide in the presence of activated carbon (9–14). Under industrial alkaline condition of gold elution, cyanide is decomposed via several reactions (11), of which the following are considered to be the most important.

Hydrolysis:

$$CN^- + 3H_2O \Rightarrow \{HCOONH_4\} + OH^- \quad (1)$$

$$\{HCOONH_4\} + \frac{1}{2}O_2 \Rightarrow HCO_3^- + NH_4^+ \quad (2)$$

$$HCO_3^- + NH_4^+ + 2OH^- \Rightarrow (\text{pH } 10.5) \Rightarrow NH_3 + CO_3^{2-} + 2H_2O \quad (3)$$

Oxidation:

$$CN^- + \frac{1}{2}O_2 \Rightarrow \{CNO^-\} \quad (4)$$

$$\{CNO^-\} + 2H_2O \Rightarrow CO_3^{2-} + NH_4^+ \quad (5)$$

The above reactions would increase the negative charge density on the surface and render it less receptive for $Au(CN)_2^-$. Although this modification of the carbon surface would occur already during the pretreatment step, the high concentration of cations (M^{n+}) would favor the formation of $M^{n+}\{Au(CN)_2^-\}_n$ ion-pairs on the carbon surface and thereby restrict the desorption of gold during this stage. Once the concentration of cations is lowered during the elution stage, the $Au(CN)_2^-$ will be desorbed from the deactivated carbon surface.

Adams (13, 14) explained that the oxidation reactions (Eqs. 4 and 5) are catalyzed by the presence of activated carbon. Nicol (10) showed that at room temperature, the decomposition of cyanide was very slow unless carbon was present. He further observed that the effect of the presence of carbon decreased with an increase in temperature. This suggests that the hydrolysis reaction is dominant at high temperatures, whereas the catalytic oxidation reaction is more significant at low temperatures. This shift in reaction mechanism occurs because of the decreased rate of the oxidation reaction at the very low dissolved oxygen concentrations (12) at high temperatures. Muir et al. (12) regarded the hydrolysis reaction as the main cause for the loss of cyanide during Zadra or AARL elution procedures. They found the rate of hydrolysis to be more temperature sensitive than the rate of oxidation, but independent of pH higher than 10.5. Measurement of the decomposition rate of cyanide revealed first-order kinetics for both the hydrolysis and oxidation reactions (11, 13).

Unfortunately, these studies have been largely empirical, with little emphasis on modeling either the kinetics of cyanide destruction or the dynamics of cyanide removal from a packed column. It is therefore the objective of the present paper to quantify the factors determining the rate of cyanide decomposition, and then to simulate the profiles of cyanide elution during the stripping of gold cyanide from a packed bed of activated carbon under different operating conditions. It will be shown that the same kinetic parameters in a model based on film transfer and first-order chemical reaction control could be used for the different experimental runs.

EXPERIMENTAL

Potassium aurocyanide and potassium cyanide dissolved in distilled water were used as the main adsorbates. Small amounts of *HCl* or *KOH*

were added manually to adjust the pH. All chemicals were of AR grade. Eluted activated carbon from a gold plant at the East Witwatersrand in South Africa was acid washed and rinsed with deionized water to remove as many impurities as possible prior to use. Unless otherwise specified, the carbon was dried at 120°C for 3 days before being weighed, soaked in water, and used in the experiments. By using the procedure described earlier (6) it was found that no AuCN was formed on the carbon during adsorption from alkaline solutions at room temperature. Unless specified otherwise, the mean particle size was 1.42 mm, which resulted in a void fraction of 0.292 in a packed column. The activated carbon had an apparent density of 837 $kg \cdot m^{-3}$, a BET surface area of 792 $m^2 \cdot g^{-1}$, and a pore volume of 6.34×10^{-4} $m^3 \cdot kg^{-1}$ which was determined by measuring the mass loss upon oven drying of a sample of carbon saturated with water.

Some tests were conducted in batch-stirred reactors containing different volumes of liquid and quantities of activated carbon. Different rates of agitation, constant temperatures, particle sizes of carbon, and initial concentrations of cyanide were used.

Most runs were conducted in a small stainless steel column packed with activated carbon in which the downward flow of eluant through the column was controlled by a peristaltic pump. Elevated temperatures were attained by operating the column under pressure and by circulating heated oil through the jacket. A PID controller manipulated both the oil flow rate and the electric current to the oil heater on the basis of the measured temperature inside the column. The column had an internal diameter of 12 mm, the bed height used was 152 mm, and the bed contained 10.2 g carbon on a dry basis. A bed volume (BV) is defined here as the empty volume of the reactor that is occupied by the packed bed of carbon, i.e., 17.19 cm^3.

Unless specified otherwise, the pretreatment step was conducted outside the column in glass beakers for 30 minutes in 20 mL of a 15.8 g KCN/L solution at 20°C. After the pretreatment the carbon was separated from the solution with a strainer, and excessive solution was removed by blotting with filter paper. The carbon was then dropped into the column and the latter filled with eluant before raising the temperature by circulating the oil through the jacket. The starting time for the elution was taken as the moment when the elution temperature was attained and the flow of eluant subsequently introduced.

KINETIC FACTORS

Figure 1 shows the effect of temperature on the decomposition of cyanide in the absence of carbon. The first-order rate constants determined

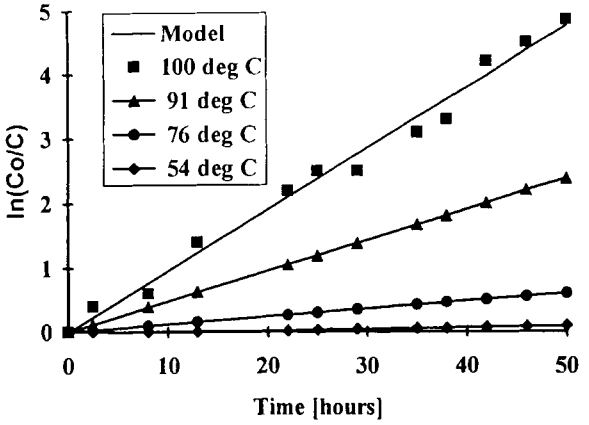

FIG. 1 The decomposition of cyanide at different temperatures in the absence of carbon. Initial concentration = 720 g CN^-/m^3. Volume of solution = 1.5 L. Stirring speed = 1400 rpm.

from the slopes of these lines are depicted in an Arrhenius plot in Fig. 2. As it is impossible to isolate the oxidation and hydrolysis reactions, the plots in Figs. 1 and 2 represent combinations of these two reactions. The regression line on the Arrhenius plot gives the following rate equation (k in s^{-1}) for the decomposition of cyanide in the liquid phase:

$$k = 4.17 \times 10^7 \exp(-87143/R_0T) \tag{6}$$

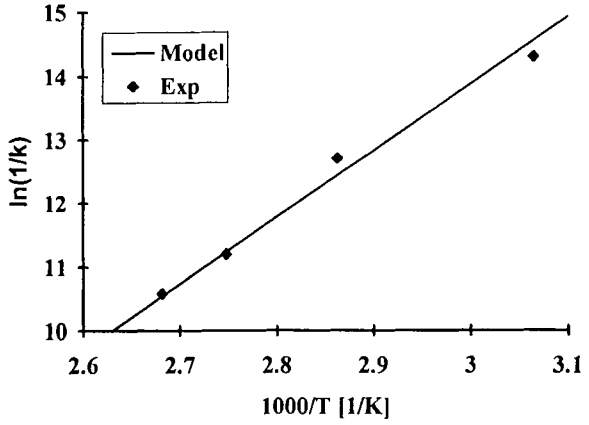

FIG. 2 Arrhenius plot of the rate constants k (s^{-1}) for the decomposition of cyanide in the absence of carbon, as derived from Fig. 1.

The activation energy of 87.14 kJ/mol compares favorably with the activation energy for the hydrolysis reaction as reported by Muir et al. (12). This implies that mainly the hydrolysis reaction occurred in the above experiments which were all conducted at temperatures higher than 50°C.

In the presence of activated carbon, Muir et al. found that the rate constant of the oxidation reaction was a direct function of the carbon concentration (12). These authors made no distinction between adsorption and oxidation of the cyanide. Adsorption could have accounted for a large percentage of the "reacted" cyanide because the experiments were conducted at room temperature to minimize hydrolysis of the cyanide. The carbon concentration and particle size were reported to have the largest influence on the oxidation of cyanide (12). Although the concentration of carbon proved to be just as important in the case of carbon used here, Fig. 3 shows that the oxidation of cyanide on coconut shell carbon is insensitive to the particle size of the carbon. The mixing speed in batch reactors had a much more prominent effect on the decomposition of cyanide in contact with the carbon, as shown in Fig. 4. The results of Figs. 3 and 4 imply that resistance to film diffusion is the rate-controlling step and that resistance to pore diffusion can be neglected (15).

Controversy exists in the literature on the relative contributions of the adsorption and the decomposition of cyanide on activated carbon to the total consumption thereof. Muir et al. (12) claim that the "poor" adsorp-

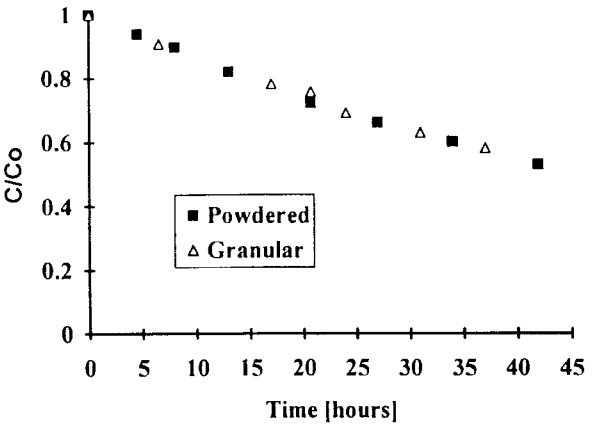

FIG. 3 The effect of particle size on the adsorption and decomposition of cyanide on activated carbon at 20°C, pH 11 and initial concentration of 404 g CN^-/m^3, with volume of solution = 1.0 L and mass of carbon = 1.50 g. Stirring speed = 1420 rpm. Particle sizes: powdered carbon = 100% −75 μm; granular carbon = +1.4–1.7 mm.

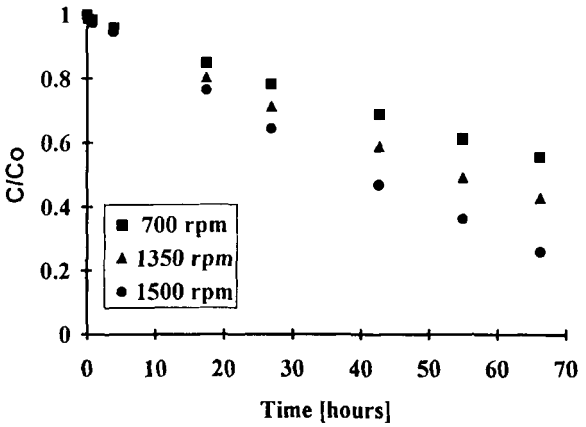

FIG. 4 The effect of mixing speed on the adsorption and decomposition of cyanide on activated carbon at 29°C, pH 11.5, initial concentration of 395 g CN^-/m^3, volume of 1.0 L, and 1.50 g carbon.

tion of CN^- at room temperature (0.1 g/kg) is in accordance with the adsorption of other simple anions like OH^- and Cl^-. Adams (14), however, attributes 35% of the cyanide loss, under the same conditions, to adsorption onto the carbon (2 g/kg). At high cyanide concentrations, as will be the case in the AARL pretreatment, he expects that the relative importance of the adsorption reaction will be reduced. Much of the disagreement on the ratio of adsorption to reaction of cyanide that is in contact with carbon can be attributed to the variation in the characteristics of activated carbons.

The elution profile of cyanide in the eluate of a Zadra elution is plotted in Fig. 5. As no electrowinning of the recovered gold was performed, the eluant was not recycled and the carbon was therefore supplied with an eluant with a constant cyanide concentration. It is evident from the profile in Fig. 5 that the conversion of the cyanide declined with time. Constant conversion of a reactant in a plug-flow reactor results in a constant concentration of the reactant in the solution leaving the reactor (15). This means that the activity of the carbon toward cyanide decreases as a result of the reaction of the cyanide. The effect of the "cyanide loading" Q of carbon on its activity toward cyanide is depicted in Fig. 6, where one sample of carbon was repeatedly contacted with "fresh" solutions with the same initial cyanide concentrations. The reaction rate constant and initial "cyanide loading" were calculated for each run from the initial and final cyanide concentrations. These reaction rate constants k_p are plotted against

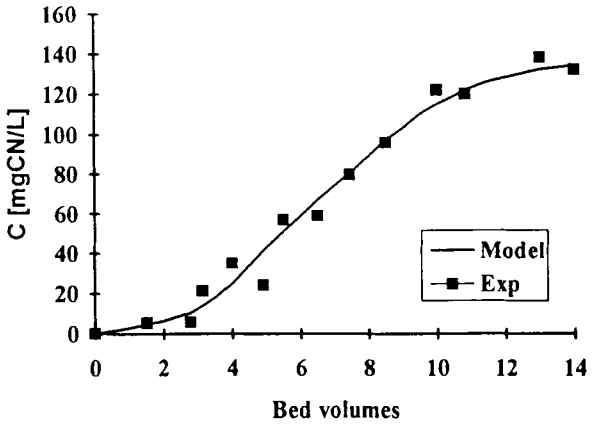

FIG. 5 Simulated and experimental cyanide profile in the eluate of a Zadra elution at 70°C, pH 10.7 and a flow rate of eluant of 47.34 mL/h containing a constant concentration of 191 g CN^-/m^3. The 10.2 g of carbon initially contained 4.78 g Au/kg and had not been pretreated.

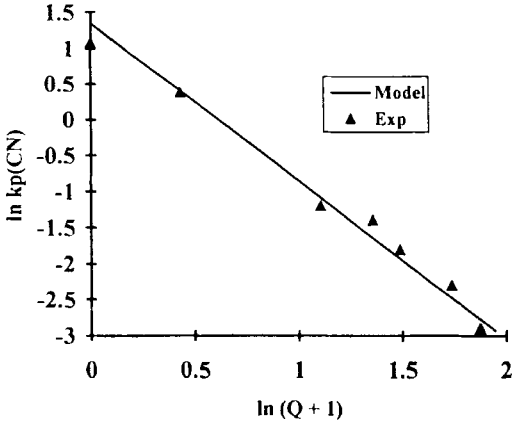

FIG. 6 The inhibiting effect of cyanide decomposed and adsorbed on the carbon ("cyanide loading") on the first-order rate constant for the decomposition of cyanide. The carbon with a mass of 21 g was contacted repeatedly for different periods with 350 mL of solution containing 0.635 g CN^-/L at 18°C and a stirring speed of 700 rpm. $k_p(CN^-)$ is given in [1/h] and Q in [g CN^-/kg].

the cumulative "cyanide loading" Q on a logarithmic scale in Fig. 6. As expected from Fig. 5, the rate of the decomposition of the cyanide decreases with an increase in the "cyanide loading" of the carbon. A linear regression of the data in Fig. 6 gives the following relationship between k_p (in 1/h) and Q (in g CN^-/kg)

$$k_p = \frac{3.85\ (h^{-1})}{(Q + 1)^{2.2}} \tag{7}$$

DYNAMIC COLUMN ELUTION MODEL

It has been shown that the uptake of cyanide is independent of carbon particle size but sensitive to mixing speed. This means that the mass transfer between the bulk solution and the carbon particles will be the rate-determining factor and that intraparticle diffusion will be of minor importance. Moreover, the rates of oxidation and hydrolysis in the bulk solution should differ from those in the pore liquid due to the catalytic role of the carbon and the deactivating effect of cyanide on the carbon. If all these subprocesses were to be considered separately, a most complex and impractical model would be required, so the following assumptions were made to simplify the proposed model.

Assumptions

1. The oxidation and hydrolysis reactions of the cyanide in the bulk solution can be combined and described by a single first-order reaction.
2. Adsorption of cyanide at gold elution temperatures is negligible (5).
3. The oxidation and hydrolysis reactions of the cyanide in the carbon pores can be combined and described by a single first-order reaction.
4. The cyanide concentration inside the carbon particles is homogeneous.
5. Mass transfer between the bulk solution and the pore liquid occurs via diffusion through a liquid layer surrounding the carbon particles.
6. The combined reaction rate in the pores of the carbon will change with the "cyanide loading" of the carbon as described by Eq. (7).

Formulation of Model

For the purposes of modeling, a packed column of carbon is considered as a series of perfectly mixed reactors of incremental height Δh with no axial or radial dispersion. Evidently, an ideal plug-flow reactor is obtained

with $\Delta h \rightarrow 0$. The following material balances could be written for the cyanide (CN^-) in each of the incremental sections.

A cyanide balance in the pore liquid is given by

$$\rho V_p \frac{\partial C_p}{\partial t} = \tilde{n} a_c - k_p C_p \rho V_p \tag{8}$$

where $\tilde{n}$, the flux of cyanide through the liquid film surrounding the particles, can be written as

$$\tilde{n} = k_s(C - C_p) \tag{9}$$

With the assumption of spherical carbon particles, the specific external area of the carbon becomes

$$a_c = 6/d_c \tag{10}$$

Substitution of Eqs. (9) and (10) in Eq. (8) yields

$$\frac{\partial C_p}{\partial t} = \frac{6k_s}{\rho V_p d_c}(C - C_p) - k_p C_p \tag{11}$$

For plug flow a mass balance of cyanide in the interparticle solution of the elution column can be written as

$$\frac{\partial C}{\partial t} = -\frac{V}{a\epsilon}\frac{\partial C}{\partial h} - \frac{6k_s(1 - \epsilon)}{\epsilon d_c}(C - C_p) - kC \tag{12}$$

The temperature dependence of the rate constant k is calculated as in Eq. (6):

$$k = b \exp(-E/R_0 T) \tag{13}$$

The type of activated carbon and the temperature both affect the rate constant k_p, which is assumed to change with the "cyanide loading" of the carbon as in Eq. (7):

$$k_p = \frac{f \exp(-E_p/R_0 T)}{(Q + 1)^g} \tag{14}$$

The change in the "cyanide loading" of the carbon is calculated from the amount of decomposition of cyanide in the pores of the carbon as follows:

$$\partial Q/\partial t = V_p k_p C_p \tag{15}$$

Equations (11) to (15) could be solved simultaneously by discretizing with backward differences in time and column height in a Turbo Pascal program.

If b and E are known from independent experiments, k_s, f, E_p, and g have to be determined empirically for a specific type of carbon at different

temperatures. Information available in the literature could be used to generate a first approximation of k_s and E_p. In this study, typical values of $k_s = 2.2 \times 10^{-6}$ m/s and $E_p = 36$ kJ/mol are used. A similar activation energy for the oxidation reaction is given by Hoecker and Muir (11). Equation (6) gives $b = 4.17 \times 10^7\ s^{-1}$ and $E = 87.14$ kJ/mol, while Eq. (7) shows that $g = 2.2$. Consequently, $f = 3100\ s^{-1}$ could be estimated from elution runs at different temperatures.

As the decomposition of cyanide had been shown to be catalyzed by activated carbon (11, 13), it was assumed that the concentration of cyanide in the pore liquid would be lower than the concentration in the interparticle solution at the end of the pretreatment stage. For modeling purposes it was found that the ratio of the former to the latter concentration was about 0.9.

EVALUATION OF MODEL

It is important to note that the same values of the parameters provided in the previous section have been used in all simulations of elution runs, except where mentioned otherwise. Figure 5 shows a simulation of the cyanide profile as measured during a Zadra elution. By continuously calculating the "cyanide loading" of the carbon, it is possible to account for the passivation of the carbon toward the decomposition of the cyanide. The simulation in Fig. 7 differs from that in Fig. 5 in that the carbon was soaked in a cyanide pretreatment solution before the introduction of the eluant. This case represents a combination of a Zadra and an AARL procedure. To prevent the calculation of a false "cyanide loading" due to dilution, the initial "cyanide loading" of the carbon was calculated from the result of an identical pretreatment conducted with dry carbon.

It is illustrated in Fig. 8 that the same parameters used for the simulation of the cyanide profiles during the Zadra process can be used for different AARL profiles. Figure 8 contains the plots of results from three different elutions which were identical as far as cyanide is concerned. These simulations illustrate the applicability of the model to describe the cyanide profiles during AARL as well as Zadra elutions. All the data shown in these figures were collected from elutions at 70°C. The simulation in Fig. 5 is repeated in Fig. 9 with $k_p = 0$ and $k_s = 0$ to show the contribution of the decomposition of cyanide in the interparticle solution at different temperatures. From this simulation it follows that decomposition in the bulk solution is negligible at 70°C and only becomes significant at 120°C. A similar simulation as in Fig. 8, but with $k_p = 0$ and $k \neq 0$, gives the same curve as in Fig. 8. This implies that little further decomposition of cyanide occurs during the elution step at 70°C after the cyanide pretreatment. From Fig.

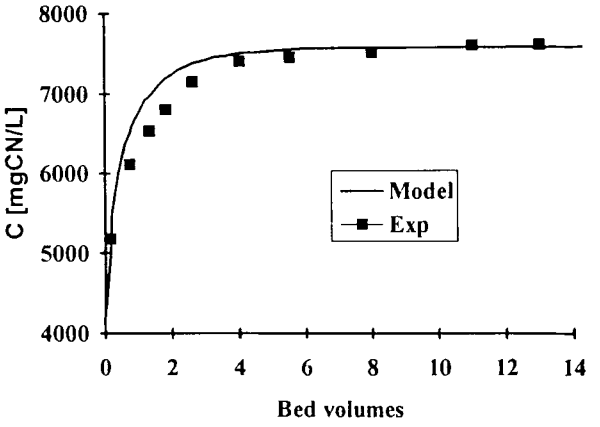

FIG. 7 Simulated and experimental cyanide profile in the eluate of a pseudo-Zadra elution at 70°C and a flow rate of eluant of 48.8 mL/h containing a constant concentration of 7726 g CN^-/m^3. The 10.2 g of carbon initially contained 4.78 g Au/kg, and had been pretreated in 20 mL of solution of 15.8 g KCN/L for 30 minutes at 20°C. After pretreatment the interparticle solution contained 4567 g CN^-/m^3 and the loading of cyanide Q = 3.437 g CN^-/kg.

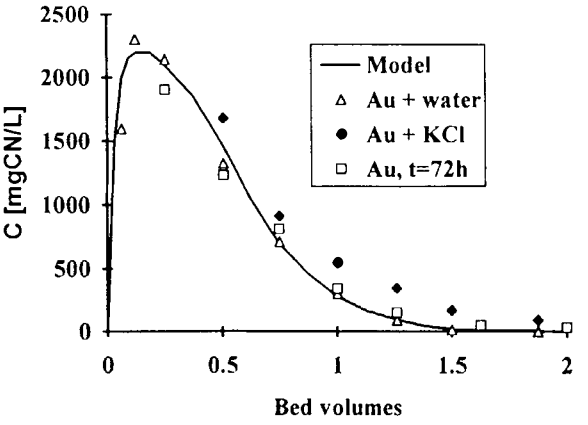

FIG. 8 Simulated and experimental cyanide profile in the eluate of an AARL elution at 70°C with either distilled water (△, □) or 560 g K^+/m^3 (◆) as eluant at a flow rate of 49.54 mL/h. The 10.2 g of carbon initially contained 4.70 g Au/kg after loading for 21 h (△, ◆) or 72 h (□), and had been pretreated in 20 mL of solution of 15.8 g KCN/L for 30 minutes at 20°C. After pretreatment the interparticle solution contained 4567 g CN^-/m^3 and the loading of cyanide Q = 3.437 g CN^-/kg.

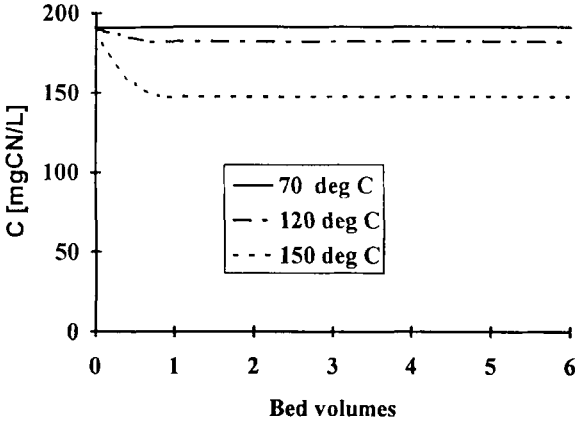

FIG. 9 Simulated contribution of the decomposition of cyanide in the interparticle solution for the Zadra elution run depicted in Fig. 5, except that $k_p = 0$ and $k_s = 0$.

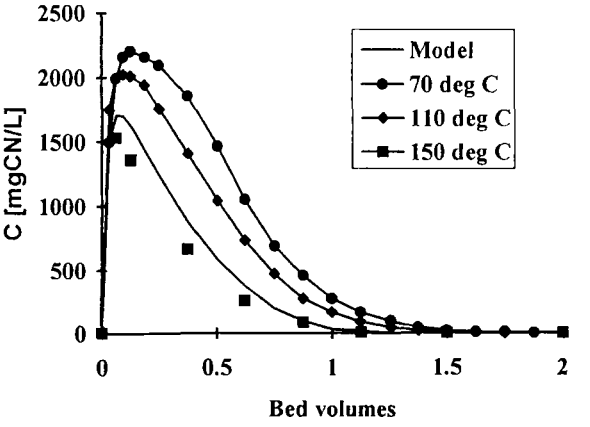

FIG. 10 Simulated and experimental cyanide profiles in the eluate of an AARL elution at different temperatures at an eluant flow rate of 48.9 mL/h. The 10.2 g of carbon initially contained 4.70 g Au/kg and had been pretreated in 20 mL of solution of 15.8 g KCN/L for 30 minutes at 20°C. After pretreatment the interparticle solution contained 4567 g CN^-/m^3 and the loading of cyanide $Q = 3.437$ g CN^-/kg.

10 it is evident that the same model parameters give satisfactory predictions of AARL elution runs conducted at different temperatures.

CONCLUSIONS

A kinetic model was proposed for the change in the cyanide concentration during the elution of gold cyanide from activated carbon in packed beds. This model was based on different first-order decomposition rates of cyanide in the interparticle solution on the one hand and inside the carbon pores on the other hand. The degree to which cyanide passivates the carbon surface is temperature dependent and is incorporated in the model, together with a mechanism of film transfer from the interparticle solution. It was demonstrated that this model is applicable to both the AARL and Zadra elution processes. This is significant because the complex AARL process has not been modeled to the same extent as the simpler Zadra process by other authors. Moreover, the same kinetic parameters could be used to simulate a wide range of operating conditions such as the level of cyanide in the eluant and different temperatures. The cyanide profile predicted by this model is essential in a generalized kinetic model for the elution of gold from activated carbon. Furthermore, the principles of this model could also be applied to processes in which cyanide is decomposed in the presence of activated carbon for environmental purposes.

SYMBOLS

a	flow area of column
a_c	specific external area of activated carbon
b	preexponential factor in Eq. (13) relating to the decomposition of cyanide in the interparticle solution
C	concentration in the bulk liquid phase
C_p	liquid phase concentration in the carbon pores
d_c	diameter of carbon particle
E	activation energy for the decomposition of cyanide in the interparticle solution
E_p	activation energy for the decomposition of cyanide in the carbon pores
f	preexponential factor in Eq. (14) relating to the decomposition of cyanide in the carbon pores
g	empirical parameters in Eq. (14) relating to the passivation of the carbon
h	height variable in a packed column

k reaction rate constant for the decomposition of cyanide in the bulk liquid
k_p reaction rate constant for the decomposition of cyanide in the carbon pores
k_s film transfer coefficient
$\bar{n}$ mass flux due to film transfer
Q loading of adsorbed and decomposed cyanide on the activated carbon
R_0 ideal gas constant
t time variable
V volumetric flow rate
V_p specific pore volume of activated carbon

Greek

ϵ void fraction in packed bed of activated carbon
ρ apparent density of activated carbon

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