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AN ANALYSIS OF THE STEADY STATE BEHAVIOR OF A MIXER-SETTLER EXTRACTION SYSTEM

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Abstract—The steady state behavior of a multi-component mixer-settler extraction system has been analyzed by adopting a mixing cell model with the individual Murphree stage efficiency parameters.

Empirical equations for the chemical equilibrium of HNO_3-$UO_2(NO_3)_2$-TBP system have been derived and incorporated into the model equations. Nonlinear Component Block Successive Relaxation (NCBSR) method executed through an Approximate Newton Raphson routine has been proposed for the solution of nonlinear steady state system equations and compared with the other methods.

The model and the derived equilibrium relations were found to be successfully employed in depicting the steady state behavior and the proposed NCBSR method was proven for its effectiveness in comparsion with the other methods.

INTRODUCTION

Mixer-settler is an equipment in wide utilization in solvent extraction. It has been one of the equipments of prime importance in the separation of unburnt uranium from spent nuclear fuels because of its operational and maintenance advantages over the other type contactors.

Since high purity is required for the uranium separated out, rather precise informations on the dynamics and the steady state behavior are of vital importance in the equipment and process design and the optimal operation, as well.

Numerous research activities have been directed toward the dynamics of mixer settler equipment systems [1.2.3].

Aly & Wittenmark [5] and Cadman & Hsu [6] have adopted a simple mixing cell model while Rozen et al.[7] and Gaudernack et al. [8] have distinguished the dynamic characteristics of a mixer and those of a settler.

Meanwhile, the concept of by-pass and Murphree plate efficiency have often been empolyed in correlating the extents of mass transfer in the process [8.9]. Murphree plate efficiency, however, can be preferably applied since the mass transfer coefficent for the individual component of different physico-chemical properties can not be described by by-pass concept, in multi-component systems. Other approaches for estimating the mass transfer coefficients by correlating with drop sizes, residence time distribution and physico-chemical properties, have been found inadequate because of the restrictions imposed on them.

The objective of this study is to analyze the steady state behavior of a mixer-settler battery system involving an extraction of uranium in HNO_3-$UO_2(NO_3)_2$ solution by tri-butyl phosphate (TBP), by mathematical modelling.

Empirical equations have been derived for the chemical equilibria between two phases and together with these equations, a mixing cell model has been adopted to analyze the steady state behavior of an 8-stage mixer-settler battery system.

For the solution of the steady state, nonlinear system equations, a new approach, a Nonlinear Component Block Successive Relaxation method has been proposed and its advantages over the other commonly employed techniques, have been shown.

THEORETICAL

1. The Chemical Equilibria

Under the assumptions of complete ionization and non-competing side reactions, the extraction of uranyl

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nitrate in nitric acid solution by TBP takes place according to the reaction schemes;

$$UO_2^{++}+2NO_3^-+2TBP_f+nH_2O \rightleftharpoons UO_2(NO_3)_2\cdot 2TBP\cdot nH_2O \qquad (1)$$

$$H^++NO_3^-+TBP_f+mH_2O \rightleftharpoons HNO_3\cdot TBP\cdot mH_2O$$

In terms of the activity, the equilibrium constants are;

$$k_U=k_U'\left(\frac{\gamma_{UO_2^{++}}\cdot\gamma^2_{NO_3^-}\cdot\gamma^2_{TBP_f}\,a^n_{H_2O}}{\gamma_{UO_2(NO_3)_2\cdot 2TBP_f\cdot nH_2O}}\right)$$

$$k_H=k_H'\left(\frac{\gamma_{H}\cdot\gamma_{NO_3^-}\cdot\gamma_{TBP_f}\,\alpha^m_{H_2O}}{\gamma_{HNO_3\cdot TBP\cdot mH_2O}}\right) \qquad (2)$$

The activity coefficients for electrolytes, however, can not be estimated by Van Laar or Margules equations and Debye-Hükel theory can be applicable primarily for dilute solutions.

Hence, an attempt was made to develope expressions for k_U and k_H in terms of the second and the third order polynomials in ionic strength, X_I;

$$k_U=a_1X_I^3+a_2X_I^2+a_3X_I+a_4$$

$$k_H=b_1X_I^2+b_2X_I+b_3$$

$$X_I=3X_U+X_H \qquad (3)$$

where X_U and X_H are the aqueous concentrations of uranylnitrate and nitric acid respectively, and a_1 ,......, and b_1,....., are constants.

From these, the expressions of distribution coefficients can be readily derived;

$$D_U=\frac{Y_U'}{X_U}=k_U(2X_U+X_H)^2Y_{T_f}^{\ 2}$$

$$D_H=\frac{Y_H}{X_H}=k_H(2X_U+X_H)Y_{T_f}$$

$$Y_{T_f}=Y_{T_o}-2Y_U-Y_H \qquad (4)$$

where Y_U, Y_H are the concentration of uranylnitrate and nitric acid in organic phase, respectively, and Y_{T_o} is the initial concentration of TBP, which is estimated by the relation;

$$Y_{T_o}=\frac{(\text{Vol. \% of TBP})\,(0.973)\,(10)}{(266.3)}\,(\text{mole}/l)$$

The concentration of free TBP, Y_{T_f} is then given by the formula

$$Y_{T_f}=\frac{1}{4k_UX_U(2X_U+X_H)^2}\Big(-(1+k_HX_H(2X_U+X_H))+\sqrt{(1+k_HX_H(2X_U+X_H))^2+8k_UX_U(2X_U+X_H)^2Y_{T_o}}\Big)$$

Hence, the distribution coefficients, D_U and D_H can be expressed as the functions of the concentrations in the aqueous phase.

2. The Model and the Steady State Balance Equations

By adopting a mixing cell model the steady state balance equations have been derived under the following assumptions;

- the interface fluctuations are negligible
- the flow rates and the hold-ups in each phase are kept constants
- the settlers are consisted of time lagging zone and a section where a partial mixing takes place
- Murphree stage efficiency remains the same in all stages
- mixing takes place in the mixer and partially in the settler

for k component in ith stage, one obtains,

$$H(X_{k\,i-1}-X_{ki})-T_{ki}=0$$

$$L(Y_{k\,i+1}-Y_{ki})+T_{ki}=0 \qquad \begin{matrix} i=1,2,\cdots,N \\ k=1,2,\cdots,K \end{matrix} \qquad (5)$$

where

$$Y^*_{ki}=D_{ki}X_{ki}$$

The mass transfer coefficient can be correlated with Murphree stage efficiency by letting $E_{ki}=E_k$ (Refer to the last assumption above)

$$E_k=\frac{L}{M_L}\,\frac{(\eta_k)}{(1-\eta_k)} \qquad (6)$$

The Murphree stage efficiency η_k can be estimated by optimizing through multi-dimensional pattern search using the formula;

$$\underset{\eta_k}{\text{Min}}=\sum_{k=1}^{K}\sum_{j=1}^{N}\{(X^E_{kj}-X^M_{kj}\eta_k)^2+(Y^E_{kj}-Y^M_{kj}\eta_k)^2\} \qquad (7)$$

where the superscripts E and M refer to the experimental and predicted values, respectively.

3. Numerical Solution—the NCBSR

The solution to the higher order, nonlinear algebraic system of equations thus far derived can be obtained by numerical technique.

False transient method (or Relaxation method) have often been employed [24] especially when the nonlinearity was severe. This methed, however, not only requires large computer capacity and time but faces error generation with increasing number of stages and components. In order to relax these problems, it has been common to employ Block Successive Relaxation [19,20,21,22,23] by partitioning the matrix stage-wise into several blocks.

Numerous discussions on this technique, by Tomas Vanek et al. [14], Kubicek & Hlavacek et al. [20] and Hageman and Porsching [21,22] appear in the literatures.

In this study, Component Block Successive Relaxation has been attempted by partitioning the matrix into Component Blocks in place of Stage Block and neglecting the Other Component Interaction (OCI) terms.

Combining the equations in(5) one obtains,

$$\frac{H}{L}(X_{ki-1}-X_{ki})+(Y_{k\,i+1}-Y_{ki})=0 \qquad (8)$$

From the relations of Murphree stage efficiency, the equilibrium line and the operating line, Y_k can be eliminated, thus obtaining,

$$f_{ki}=\frac{H}{L}X_{ki-1}-(\eta_k D_{ki}+(2-\eta_k)\frac{H}{L})X_{ki}+(\eta_k D_{ki+1}$$
$$-(1-\eta_k)\frac{H}{L})\ X_{ki+1}=0,\quad i=1,2,\cdots,N-1$$
$$f_{kN}=\frac{H}{L}X_{kN-1}-(\eta_k \dot{D}_{kN}+\frac{H}{L})X_{kN}-\eta_k Y_{kN+1}=0 \quad (9)$$

Arranging in Component Blocks, these equations can be expresed in matrix notation.

$$\underline{Z}_k^T=(X_{k1},X_{k2},\cdots,\ X_{kN})$$
$$\underline{Z}^T=(\underline{z}_1^T,\underline{z}_2^T,\cdots,\ \underline{z}_k^T)$$
$$\underline{f}_k^T=(f_{k1},f_{k2}\cdots,\ \underline{f}_{kN})$$
$$\underline{F}^T=(\underline{f}_1^T,\underline{f}_2^T,\cdots,\ \underline{f}_k^T)\quad \text{and}$$
$$\underline{F}(\underline{Z})=\underline{0} \quad (10)$$

Taking the first term from the Taylor serices expansion of this equation, and arranging in the form according to Newton-Raphson method one obtains,

$$\underline{F}(\underline{Z}^{m+1})=\underline{F}(\underline{Z}^m)+\underline{\underline{J}}^m\Delta\underline{Z}^m=\underline{0}$$
$$\text{where } \Delta\underline{Z}^m=\underline{Z}^{m+1}-\underline{Z}^m \quad (11)$$

The Jacobian matrix, $\underline{\underline{J}}^m$ is,

$$\underline{\underline{J}}m=(\frac{\partial f}{\partial \underline{Z}})_{\underline{Z}=m}=\begin{bmatrix}\frac{\partial \underline{f}_1}{\partial \underline{Z}_1} & \cdots\cdots & \frac{\partial \underline{f}_1}{\partial \underline{Z}_k}\\ \vdots & & \vdots\\ \frac{\partial \underline{f}_k}{\partial \underline{Z}_1} & \cdots\cdots & \frac{\partial \underline{f}_k}{\partial \underline{Z}_k}\end{bmatrix} \quad (12)$$

Therefore,

$$\underline{\underline{J}}^m\Delta\underline{Z}^m=-\underline{F}^m=-\underline{F}(\underline{Z}^m)$$
$$\underline{Z}^{m+1}=\underline{Z}^m-(\underline{\underline{J}}^m)^{-1}\underline{F}(\underline{Z}^m)$$

Rewriting the equation in the terms for Self Component Interaction (SCI) and for Other Component Interaction, it new has the expression

$$\underline{Z}_k^{m+1}=\underline{Z}_k^m-(\underline{\underline{J}}_{kk}^m)^{-1}(\underline{f}_k^m+\sum_{\substack{j=1\\ j\neq k}}^{N}=kj\ J^m\ \Delta\underline{Z}_j^m\ )$$
$$k=1,2,\cdots\cdots,\ K \quad (14)$$

In general the OCI terms are negligible as compared with the SCI terms. Hence, neglecting the OCI terms the equation (14) is now,

$$\underline{Z}_k^{m+1}=\underline{Z}_k^m-(\underline{\underline{J}}_{kk}^m)^{-1}-f_k^m$$
$$k=1,2,\cdots\cdots,K \quad (15)$$

where $\underline{\underline{J}}_{kk}$ is a tri-diagonal matrix.

The approximate solution obtained by solving equation (4) can now be corrected by applying a weighting factor, i.e.

$$\overset{*}{\underline{Z}}{}^{m+1}=\underline{Z}^m+\omega\ (\underline{Z}^{m+1}-\underline{Z}^m)\quad \text{and}$$
$$\underset{\omega}{\text{Min}}(\underline{F}^T(\underline{Z}^{*m+1})\cdot\underline{F}(\underline{Z}^{m+1}))\rightarrow\underline{Z}^{*m+1} \quad (16)$$

The calculation is then repeated until the following criteria are satisfied.

$$\underset{i}{\text{Max}}\ (\underline{Z}_{ki}^{*m+1}-\underline{Z}_{ki})\leq\varepsilon\, k$$
$$k=1,2,\cdots\cdots,K \quad (17)$$

Applying the Approximate Newton Raphson instead of Newton Raphson method by arranging the matrix into tri-diagonal form, neglecting the OCI terms, it was expected that the NCBSR was advantageous over NSBSR in the computing time, in particular. All three largely employed methods, i.e. the linearization, the optimal weighting and the fixed weighting methods have been tested and compared.

EXPERIMENTAL

1. Experimental Set-up and the Operation

The experimental set-up consisting of an 8-stage mixer-settler operated in counter-current contact between two phases, is shown in Fig. (1)

In order to keep the constant flow rates the fluid in the head control chambers have been maintained at constant level by valves, and precision syringe pumps have been used for feeding. A single motor with its speed controlled by D.C variable voltage cotrol system, has driven all the impellers.

Listed in Table 1 are the physical dimensions of the mixer-settler, the hold-ups estimated from the analysis of residence time distribution and the other conditions. The interphase was maintained at 3.2 cm from the bottom.

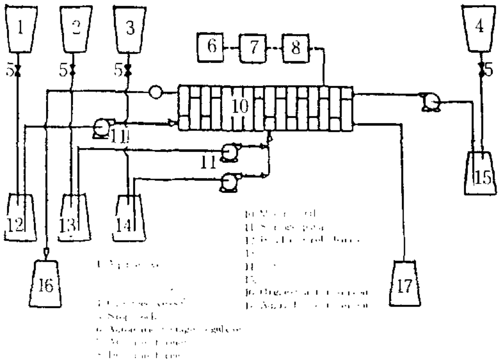

Fig. 1. Schematic flow diagram of experimental apparatus.

Table 1.

	Volume (c.c)	Hold-up total (c.c)	aq (%)	org (%)	Dead Space (c.c)
Mixer	12	12	-	-	-
Settler	36.3	34.1	0.601	0.399	2.2

Table 2. Operating Conditions.

Run No.	Feed Composition				Flow Rate		RPM	S. L. (cm)	Operating Time (hr)
	AQ		Org.		(ml/hr) AQ	(ml/hr) Org.			
	U (g/l)	H^+(N)	U (g/l)	H^+(N)					
1	48	3. 076	0. 0	0. 0	354	180	2400	3. 2	8
2	48. 5	3. 082	0. 0	0. 0	354	205. 5	〃	〃	8
3	48	3. 025	0. 0	0. 0	355	178	〃	〃	8
4	38. 5	2. 595	0. 056	0. 005	359. 3	178. 6	〃	〃	9
5	59. 8	3. 0	0. 056	0. 005	359. 3	178. 6	〃	〃	9

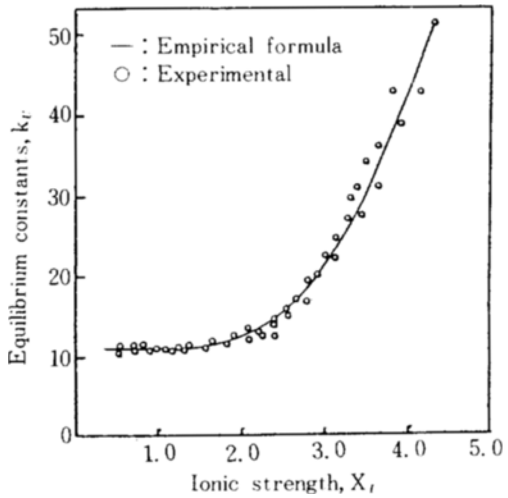

Fig. 2. Equilibrium constant of uranium vs. ionic strength.

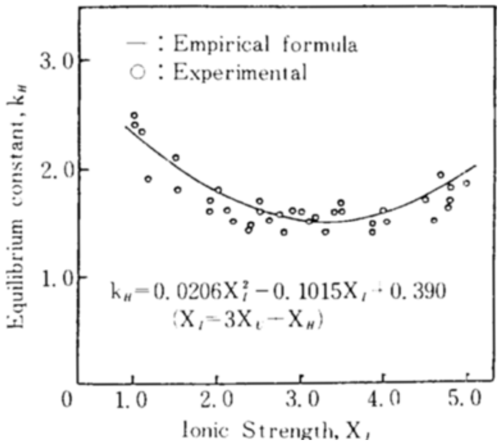

Fig. 3. Equilibrium constant of nitric acid vs. ionic strength.

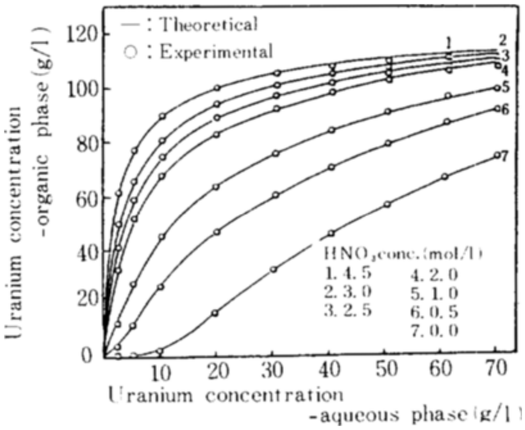

Fig. 4. Uranium distributions-at constant nitric acid concentration.

Steady state has been attained at 8 hours of operation, (Refer to the operating conditions in Table 2.) and 3 ml samples for each phase were taken from each stage for the analysis.

2. Analysis

Uranium contents were analyzed by an oxidation-reduction titration method for the contents higher than 20g/*l* and thiocyanate method was used when the contents were in the range lower than 20g/*l*.

Oxalate method was used to measure the acidity. (Shown in Figures 4 and 5 are the distribution of uranium and nitric acid in two phases predicted using these equations and compared with the experimental data.)

RESULTS AND DISCUSSIONS

1. Chemical Equilibrium

Empirical relations derived in terms of the ionic strength for the chemical equilibrium constants k_U and k_H of HNO_3-$UO_2(NO_3)_2$-30% TBP in dodecane system, are shown in Figures 2. and 3.

$$k_U = 0.8905X_I^3 - 1.2107X_I^2 - 1.3978X_I + 12.8$$

$$k_H = 0.0206X_I^2 - 0.1015X_I - 0.390$$

where $X_I = 3X_U + X_H$

Shown in Figures. 4 and 5 are the distributions of uranium and HNO_3 in two phases predicted using these equations and compared with the experimental data.

As noticed, the predictions were satisfactory at low concentration (acidity 1.5 N, uranium concentration 60 g/l). Higher predictions beyond these ranges were possibly due to the following reasons;

- incomplete ionization of the compounds
- interactions among the components affecting the activity coefficients

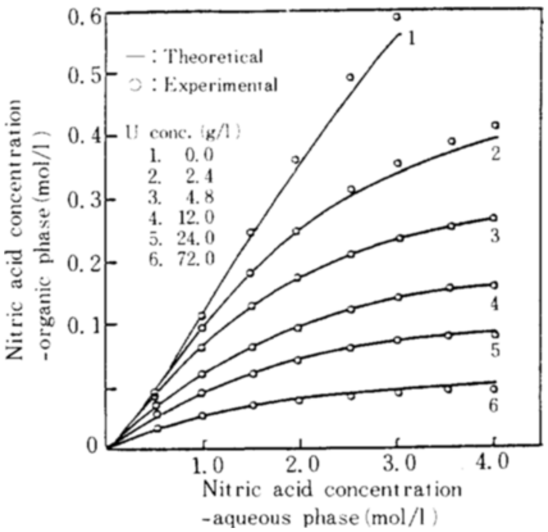

Fig. 5. Nitric acid distribution -at constant uranium concentration.

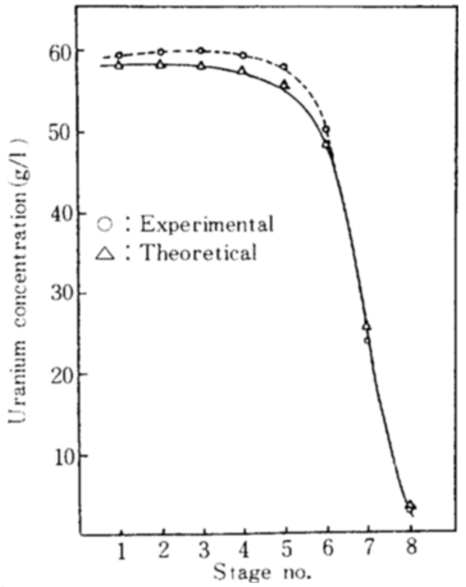

Fig. 6. Uranium distribution (aqueous phase) (Run No. 5).

2. Steady state concentration distribution

The stady state concentration distribution has been calculated on IBM 360/160 computer system. The Murphree stage efficiencies for each component has been estimated by multi-dimensional pattern optimization search.

The estimated steady state concentration distributions were compared with the experimental data in Figures. 6 through 9. the results were satisfactory within

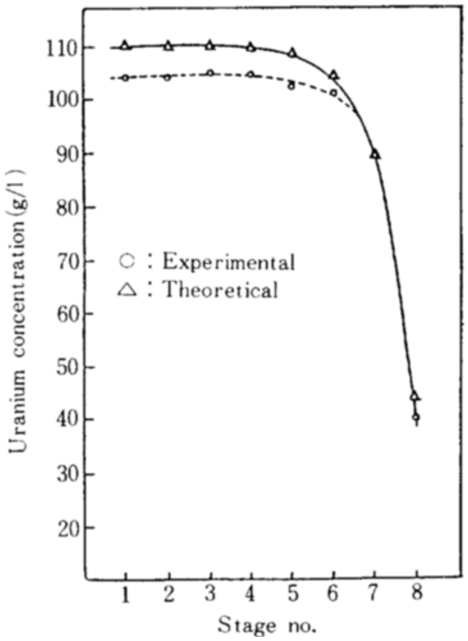

Fig. 7. Uranium distribution (organic phase) (Run No. 5).

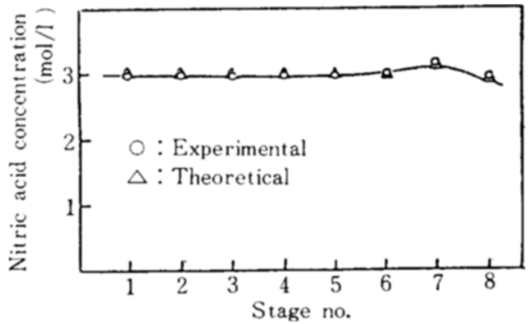

Fig. 8. Nitric acid distribution (aqueous phase) (Run No. 5).

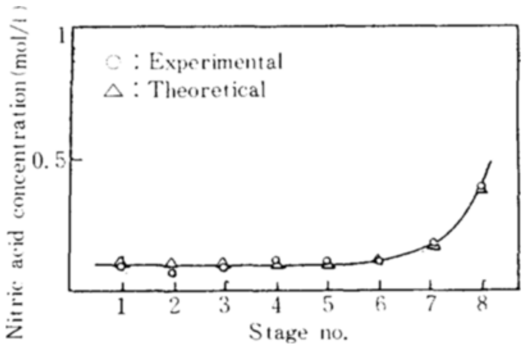

Fig. 9. Nitric acid distribution (organic phase) (Run No. 5).

the experimental error range.

3. Numerical Solution-Nonlinear Component Block Successive Relaxation (NCBSR)

The steady state concentration distribution were calculated by NCBSR, and the following results were observed; Refer to Table 3.

(1) the linearization method requiring larger amount of repeat calculations renders a conversion problem near a pinch point, in particular.

(2) the convergence has been satisfactory in both weighting methods and thus the fixed weighting method was found advantageous over the optimal weighting method in computing time.

(3) the NCBSR by fixed weighting has been shown most effective in computing the steady state concentration distribution involving two components system.

Table 3.

Exp.	NBSR		NCBSR					
			LCBSR		NCBOSR		NCBSR	
No.	No. iter	Max. error	No. iter	Max. error	No. iter	Max. error	No. iter	Max. error
1	6	3.0×10^{-4}	27	1.0×10^{-3}	10	2.2×10^{-4}	6	2.1×10^{-4}
2	8	5.0×10^{-5}	23	1.2×10^{-3}	8	1.1×10^{-4}	7	5.0×10^{-5}
3	7	7.5×10^{-5}	28	1.0×10^{-3}	11	6.1×10^{-5}	6	7.3×10^{-5}
4	6	10×10^{-5}	19	1.0×10^{-3}	8	1.5×10^{-4}	6	9.9×10^{-5}
5	10	4.3×10^{-5}	100	No. converge	8	6.7×10^{-4}	11	3.8×10^{-5}

(1) Initial Condition

$X_{Ui}=0$

$X_{Hi}=0$ $\quad i=1,2,\cdots\cdots,8$

(2) LCBSR (Linearized Component Block Successive Relaxation)
weighting factor = 1 (fixed)
NCBOSR (Nonlinear Component Block Optimal Successive Relaxation)
weighting factor: 0.0-1.5 (Golden Ratio Search)
NCBSR (Nonlinear Component Block Successive Relaxation)
weighting factor = 1 (fixed)

(3) Computer: IBM 360/160 series

CONCLUSIONS

Steady state behavior of a mixer-settler battery involving an extraction in HNO_3-$UO_2(NO_3)_2$ -30% TBP in Dodecane system, has been analyzed by mathematical modelling, and by employing a new method, the solution for a nonlinear system of algebraic model equations, has been obtained.

The following conclusions were drawn;

1. The empirical equations for the chemical equilibria have been derived and found successfully applicable.

$$Y_U=k_UX_U(2X_U+X_H)^2Y_{T_f},$$

$$Y_H=k_HX_H(2X_U+X_H)^2Y_{T_f} \text{ with}$$

$$k_U=0.8905X_I^3-1.2107X_I^2-1.3978X_I+12.8,$$

$$k_H=0.0206X_I^2-0.1015X_I-0.390 \text{ and}$$

$$X_I=3X_U+X_H$$

2. A mixing cell model with Murphree stage efficiency parameters for the individual components has been found employable for the prediction of steady state concentration distribution in the system.

3. A Nonlinear Component Block Successive Relaxation proposed in this study has been compared with other method and proven more effective for the solution of nonlinear system of algebraic equations derived for a multi-component and multi-stage equipment system.

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NOMENCLATURE

Latin Symbols

a_i, b_i : polynomial const. of equilibrium (—)
D_U, D_H: distribution coeff. of U and HNO_3 (—)
D_{ki} : ith stage k th component equilibrium constant (—)
E_{ki} : ith stage k th component mass transfer coefficient (min^{-1})
$\underline{F}$: total steady state balance eqn. vector (—)
f_{ki} : ith stage k th component steady state balance eqn. (—)
H : flow rate of heavy phase (l/min)
$\underline{\underline{J}}^m$: Jacobian matrix of m th iteration (—)
k_U, k_H : equil. const of U and HNO_3 (—)
L : flow rate of light phase (l/min)
M_L : mixing volume of light phase (l)
$\underline{O}$: null vector (—)
T_{ki} : ith stage k th component mass transfer rate between two phases (mole/min)
X_{ki}, Y_{ki}: ith stage k th component conc. heavy and light phase, respectively(mole/l)
X_{ki} : equilibrium conc. of light phase (mole/l)
X_I : ionic strength (mole/l)
X : conc. in aq. phase (mole/l)
X^E_{kj}, Y^E_{kj} : expt. conc. of jth stage k th component of aq. and org. phase (mole/l)
X^M_{kj}, Y^M_{kj} : theoretical conc. of jth stage k th component of aq. and org. phase (mole/l)
Y : conc. in org, phase (mole/l)
Y^*_{ki} : equilibrium conc. of heavy phase (mole/l)
Y_{T_o} : initial conc. of TBP in org. phase (mole/l)
Y_{T_f} : conc. of free TBP in org. phase (mole/l)
$\underline{Z}$: aqueous component vector (—)
$\underline{Z}^m$: $\underline{Z}^{m+1} - \underline{Z}^m$ (—)
$\underline{Z}^{*m+1}$: modified value $\underline{Z}^{m+1}$ by weighting factor (—)

Greek Symbols

ε : error criteria; aqueous hold-up fraction
η_k : Murphree efficiency of k th component
ω : weighting factor

Subscripts

k : k th component
i,j : i th, j th stage
N : total number of stages
k : total number of components
U : uranium
H : nitric acid
T : TBP
T : transpose
m : m^{th} iteration

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