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SELECTIVE METAL ION EXTRACTION FOR
MULTIPLE ION LIQUID-LIQUID EXCHANGE REACTIONS

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Abstract

The research in hydrometallurgical solvent extraction is to develop a fundamental means to predict selectivity during simultaneous solvent extraction of multiple metal ions when the kinetic rates and thermodynamic equilibria both do not favor the desired metal.

The first phase of selecting a model binary system to study was completed. The system selected is Cu(II), Fe(III) acid sulfate solutions extracted by β -alkenyl 8-hydroxy quino- line (Kelex 100) in xylene. Maximum copper extraction occurs in less than 5 minutes at 30-50°C.

Thermodynamic chemical equilibrium studies with the Fe(III) ion indicate that the ionic charge of the extracted ion is +3 over a limited pH and concentration range. A simplified equilibrium model did not fit the experimental data. A chemical equilibrium model for the aqueous phase is developed and will be employed to analyze existing and future data. A similar model will be employed for the Cu(II) system.

Kinetic studies on the liquid jet recycle reactor are underway. Approximately 70 experiments are planned over the composition-temperature space of $[\text{Fe}]_T$ 0.005-0.02M; $[\text{Cu}]_T$, 0.005-0.02M; $[\text{HR}]$, 0.02-0.06M; pH 0-2; and T, 25-50°C.

The model proposed to analyze simultaneous extraction of Cu(II) and Fe(III) in a stirred tank extractor was reduced to a set of two nonlinear algebraic equations for idealized kinetic expressions.

The one liter continuous flow stirred tank system for

the extraction studies is modified and shakedown of the apparatus is underway.

Introduction

The D.O.E. sponsored research in hydrometallurgical solvent extraction is to develop a fundamental means to predict selectivity during simultaneous solvent extraction of multiple metal ions. The approach is to understand the interplay between mass transfer with interfacial chemical reaction and chemical equilibria for multiple metal systems. The results will provide a rational approach for the design of multiple metal solvent extraction processes.

A four point program was proposed and is currently being pursued. These objectives are:

- Selection of a model system.
- Equilibrium studies to obtain distribution data for thermodynamic models.
- Experimental chemical reaction studies to obtain a kinetic model.
- Comparison of predictions of selectivity with experimental results for single stage contactors and analysis of multiple stage operation.

The work to date shows progress on all the above points. A summary of these results follows.

Progress of Research

The project is progressing well in that the reaction system has been identified, partial chemical equilibria data have been obtained and partially analyzed, the kinetic experimental program is defined and runs are in progress, upgrading of the one liter reactor for kinetic flow studies is near

complete, and a preliminary reactor model based on possible reaction kinetics is being tested.

(1) Selection of Model System

The model system selected should exhibit the interplay between kinetics and chemical equilibria in determining the selectivity of a metal ion in competitive extraction or stripping reactions. The three principle reactions involved can be represented by the mass action expressions

Here M^{+m} and N^{+n} are metal ions with charge numbers m and n , MR_m and NR_n are complexes, HR is a monoacidic bidentate chelating agent, and o and A refer to the organic and aqueous phases. The model system selected should meet the following requirements in the case of extraction: (a) the time scale of the forward reactions of reactions (1) and (2) should be of the same order of magnitude; (b) the forward or reverse reaction of reaction (3) should be of the same time scale as the forward reactions of equations (1) and (2); (c) the time scales of the reactions should fall within the limitations of the experimental reactor used for the study; (d) the system should be well defined, for example the physical properties and purity of the chelating acid should be known, and (e) analytical measurements should not present significant problems.

Also to be considered is the availability of physical properties, thermodynamic data, chemical equilibria data and kinetic data. This information will minimize background data acquisition.

The binary metal systems studied as candidates for the selectivity analysis are listed in Table 1. Systems 1 and 3 were studied whereas system 2 was discarded due to possible reactions in aqueous phase.

Extraction experiments were conducted in an Eberback 6250 Shakerbath with equal volumes of aqueous phase solutions of $\text{Fe}_2(\text{SO}_4)_3$ and $\text{Cu}(\text{SO}_4)$ and organic solutions of chelation acid. Reagent chemicals are employed and the chelation acid solutions are washed thrice each with acid solutions and distilled water. Analysis of metal ions is by atomic absorption spectroscopy using a Perkin-Elmer 460 A.A. Spectrophotometer.

The results of representative extraction experiments are shown in Table 2. It is observed for experiments 1 and 2 that the ligand exchange reaction is not observed as copper complex is not displaced by iron for the LIX64N system. Experiments 3 through 7 show that the copper complex is displaced by iron. A maximum in Cu extraction is observed for experiment 3 at 0.5 hours. As temperature increases the maximum copper extracted occurs in less than 5 minutes. Figure 1 compares long term extraction of copper only, iron only and the mixture of copper and iron from aqueous sulfate solutions by Kelex 100 in xylene at 24°C. The maximum concentration of copper as a result of the liquid exchange reaction occurs at about 1 hour.

TABLE 1

BINARY METAL EXTRACTION SYSTEMS STUDIED

AQUEOUS PHASE	ORGANIC PHASE	COMMENT
1. Cu(II), Fe(III), H ₂ SO ₄	β-hydroxy benzophenone oxime and α-hydroxy oxime mixture (LIX64)-toluene solvent	Liquid exchange mechanism not significant. Discarded.
2. Cu(II), Fe(III), H ₂ SO ₄	acetylacetone - CCl ₄ , chloroform, benzene or xylene solvent	Acetylacetone partially soluble in aqueous phase. Reaction may occur in aqueous phase. Discarded.
3. Cu(II), Fe(III), H ₂ SO ₄	β-alkenyl 8-hydroxy quinoline-kerosine, toluene, or xylene solvent.	Liquid exchange mechanism observed. Xylene selected as solvent. Acceptable.

For longer contact times copper is displaced by iron and a reversal of copper flux occurs. It is noted that equilibrium was attained in about 30 min. and 18 hours for the copper and iron systems respectively. However, at least 27 hours is needed for the copper iron mixture to reach equilibrium. Figure 2 shows results an extraction of copper and iron mixture over a period of 118 hours. Equilibrium occurred after about 35 hours.

It is concluded from these studies that the Cu(II), Fe(III) acid sulfate solutions extracted by β -alkenyl 8-hydroxy quino- line (Kelex 100) in xylene is a suitable system to be studied in the proposed research. Further at higher temperatures (30-50°C) the maximum extraction of copper occurs in less than 5 minutes. Xylene is selected as a solvent because it prevents sulfuric acid extraction by Kelex, it has low toxicity, and volatility and equilibrium data is available with this system.

(2) Thermodynamic Chemical Equilibrium Data.

The analysis of the chemical equilibria is needed to model the kinetics of the reaction, predict the distribution of metal ions and predict the selectivity at equilibrium. Since the Cu(II), Fe(III) sulfate solutions extracted by Kelex in xylene is the selected system, equilibrium data for Cu(II) sulfate solutions with Kelex 100 in xylene is available (1). The equilibrium studies are centered on the Fe(III), sulfate solutions with Kelex 100 in xylene. The purpose of the study is to gather data which will permit the determination

TABLE 2

EXTRACTION EXPERIMENTS TO DETERMINE LIGAND EXCHANGE REACTION

	ORGANIC PHASE	AQUEOUS PHASE	INITIAL pH	T, °C	% METAL EXTRACTED VERSUS TIME (hr)									
					Time	0.033	0.083	0.167	0.25	0.50	1.0	2.0	5.0	25.0
1	LIX64N ^a in Toluene, 1.03M	Cu(II), 0.079M Fe(III), 0.073M	2.08	24 - 25	Cu(II)	54.7	98.5	--	99	--	99	--	99	99
					Fe(III)	7.8	7.3	--	14.6	--	19.5	--	20	20
2	LIX64N in Toluene, 0.25M	Cu(II), 0.088M Fe(III), 0.158M	2.08	24 - 25	Cu(II)	--	70	--	70.8	71.7	70.8	70.8		
					Fe(III)	--	0	--	1.2	2.4	0.0	0.0		
3	Kelex 100 ^b in Toluene, 0.096M	Cu(II), 0.063M Fe(III), 0.072M	2.08	22	Time		0.083	0.167		0.50	1.0	1.5	3.0	6.0
					Cu(II)	--	44.5	45	--	46.7	44.5	43.0	40.0	35.2
4	Kelex 100 in Toluene, 0.096M	Cu(II), 0.063M Fe(III), 0.072M	2.08	32.8	Fe(III)	--	0.75	1.5	--	5.8	6.5	7.2	13.0	16.2
					Cu(II)	--	46.0	45.5	--	40.7	40.0	38.0	36.0	30.0
5	Kelex 100 in Toluene, 0.096M	Cu(II), 0.063M Fe(III), 0.072M	2.08	50	Fe(III)	--	1.75	2.5	--	4.0	7.2	9.0	13.5	17.0
					Cu(II)	--	48.4	44.5	--	41.6	37.3	--	--	24.7
6	Kelex 100 in Xylene, 0.096M	Cu(II), 0.063M Fe(III), 0.072M	2.08	32.8	Fe(III)	--	0.5	8.3	--	8.8	12.0	--	--	16.2
					Cu(II)	--	48.0	46.6	--	45.7	44.6	--	40.1	27.0
7	Kelex 100 in Xylene, 0.096M	Cu(II), 0.063M Fe(III), 0.072M	2.08	50	Fe(III)	--	1.6	2.7	--	4.5	7.5	--	14.3	16.9
					Cu(II)	--	46.7	44.2	--	40.8	36.9	--	--	26.0
7	Kelex 100 in Xylene, 0.096M	Cu(II), 0.063M Fe(III), 0.072M	2.08	50	Fe(III)	--	6.4	7.6	--	7.8	11.8	--	--	15.9

a) LIX64N is 20:1 parts by weight p-hydroxy benzophenone oxime to α -hydroxy oxime supplied by General Mills.

b) Kelex 100 is a β -alkenyl 8-hydroxy quinoline supplied by Ashland Chemicals.

of the ionic charge of the extracted iron ion and the establishment of a satisfactory thermodynamic equilibrium model.

Ionic Charge

The first approach is to employ a simple stoichiometric model for the chemical equilibrium as:

with

$$K_{\text{eq}} = \frac{[\text{FeR}_n^{+(3-n)}]_{\text{O}} [\text{H}^+]_{\text{A}}^n}{[\text{Fe}^{+3}]_{\text{O}} [\text{HR}]_{\text{A}}^n}$$

n will be 3 if only Fe(III) is extracted. Values less than 3 indicate other forms of iron complexes are extracted such as FeHSO_4^{+2} , FeSO_4^{+1} , FeOH^{+2} , etc. If $D = [\text{FeR}_n^{+(3-n)}]_{\text{O}} / [\text{Fe}^{+3}]_{\text{A}}$, then

$$\log D = \log K_{\text{eq}} + n \log [\text{HR}] + n \text{ pH} \quad (5)$$

When $[\text{HR}] \gg [\text{Fe}^{+3}]$ and the ionic strength is held relatively constant, then K_{eq} will not vary significantly. A plot of $\log D$ versus pH will give the slope n which permits estimation of the ionic charge of the average iron complex extracted.

Equilibrium experiments were conducted by contacting equal volumes of aqueous phase containing ferric ion with an organic phase of Kelex 100 in xylene. The Kelex 100 was washed three times each with 1M sulfuric acid and 1M distilled water. The extractions were carried out at 23-24°C. Atomic absorption spectroscopy was used to determine the concentration of iron in the aqueous phase. The organic phase iron concentration was calculated.

$\text{Fe}_2(\text{SO}_4)_3$ and Kelex concentrations were of the range 0.008M. and 0.5M respectively and pH varied between 0.16 to 1.04.

The results of these experiments plotted in Figure 3 show that within the pH range of 0.16 to 0.61 the slope is 3.04. This result suggests that Fe(III) is being extracted under these conditions. These results are far from complete and limited to narrow composition ranges.

Equilibrium Model for Fe(III) - sulfate-Kelex 100/xylene

A thermodynamic based model was proposed (2) to model single metal extraction equilibria of Fe(III) or Cu(II) from sulfate solutions by Kelex 100 in xylene. These models can then be used to model multiple metal equilibria. As discussed, it is necessary to determine the concentration-based equilibrium constants

$$K_{\text{Fe}} = \frac{[\text{FeR}_3]_{\text{O}} [\text{H}^+]_{\text{A}}^3}{[\text{Fe}^{+3}]_{\text{A}} [\text{HA}]_{\text{O}}^3} \quad (6)$$

$$K_{\text{Cu}} = \frac{[\text{CuR}_2]_{\text{O}} [\text{H}^+]_{\text{A}}^2}{[\text{Cu}^{+2}]_{\text{A}} [\text{HR}]_{\text{O}}^2} \quad (7)$$

and

$$K_{\text{Fe,Cu}} = \frac{[\text{Fe}^{+3}]_{\text{A}}^2 [\text{CuR}_2]_{\text{O}}^3}{[\text{Cu}^{+2}]_{\text{A}}^3 [\text{FeR}_3]_{\text{O}}^2} \quad (8)$$

where the brackets represent concentrations. The concentration-based equilibrium coefficients of each reaction is the product of the thermodynamic equilibrium constant divided by the appropriate ratio of the activity coefficients of the reactants and products raised to the power of the corresponding stoichiometric

coefficients. Appropriate values of K_{Fe} , K_{Cu} , $K_{Fe, Cu}$ are necessary so the thermodynamic models postulated (See eqns. 8-10; ref. 2) can be employed to determine the metal distribution between the phases at equilibrium.

A first level equilibrium model for determination of the concentration-based equilibrium coefficient for the Fe(III)-sulfate - Kelex 100/xylene system was employed to analyze equilibrium data. The model assumes complete dissociation of ferric sulfate into Fe(III) ions, complete dissociation of H_2SO_4 into the sulfate ion and H^+ , only monomeric forms of HR exist, and iron is extracted as Fe(III).

Equation (6) was tested with 19 data sets of equilibrium data for initial molar concentrations of total iron, Kelex and hydrogen ion in the ranges of 0.019 - 0.081, 0.057 - 0.135, and 0.0 - 0.18 respectively. The data were obtained at 23°C after 24 hours of contact. Analysis was by atomic absorption spectroscopy. The above model was found inadequate as K_{eq} varies from 2.9 to 135 with mean as 47 and standard deviation of 44.

The inadequacy of equation (6) to represent the data can be due to the inaccurate model of the aqueous phase equilibria giving rise to incorrect values of $[Fe^{+3}]$ and $[H^+]$. Also, the assumptions of invariant activity coefficients may be invalid. Accordingly, a thermodynamic model is being developed to calculate the concentrations of free Fe(III) and H^+ in the aqueous phase. The values of Fe(III) and H^+ obtained can be employed with equation (6) to determine K_{Fe} .

The aqueous phase model assumes that the following

equilibria exist:

It was determined that equations 9, 11, 13, 16 and 17 could be assumed completely dissociated for the range of concentrations of interest and for the above values of the equilibrium coefficients and stability constants. Algebraic manipulation of the resulting equations gives rise to the third order polynomial equation

$$a_3 S^3 + A_2 S^2 + a_1 S + a_0 = 0 \quad (18)$$

*CD - completely dissociated.

+ - Equilibrium constants obtained from reference (3).

where

$$S = [SO_4^{-2}]$$

$$a_3 = 1.5 \times 10^6$$

$$a_2 = 1.51 \times 10^4 + 1.5 \times 10^6 (S_T - F_T)$$

$$a_1 = 1 - 1.49 \times 10^4 S_T - 400 F_T$$

$$a_0 = F_T - S_T$$

F_T = total iron in aqueous phase

S_T = total sulfur in aqueous phase

Equation (18) can be solved for $[SO_4^{-2}]$ and subsequently employed to calculate all the aqueous phase concentrations. These calculated concentrations depend upon knowledge of total sulfate and iron concentration in the aqueous phase and the stability and equilibrium constants.

Additional equilibrium experiments are being conducted and will be analyzed according to the above outlined method to compare calculated and experimental values of K_{Fe} . If the results are satisfactory, a similar model will be developed for K_{Cu} . Otherwise, a semi-empirical approach will be employed to model the equilibria of the single metal system.

It appears that the multiple metal equilibria studies will be deleted from the program unless the single metal equilibria studies can be completed before July 15, 1980. Preliminary experiments show that (a) the optimum batch extraction time for CuR_2 during multiple metal extraction is

about 0.5 - 1.0 hours at 24°C (less than 5 minutes at 40°C), and (b) the time needed to attain equilibrium between an aqueous sulfate a mixture of Fe(III) and Cu(II) with Kelex 100/xylene is of the order of 24-30 hours. Therefore, the multiple metal equilibria data are not needed for the kinetic study as the kinetic region of interest for multiple metal extraction is far from equilibrium. The individual equilibria data are needed to determine the correct ionic charge of the extracted metal species.

(3) Kinetic Studies on the Liquid Jet Recycle Reactor

An experimental program has been developed to obtain the kinetic data for the rate of extraction of Cu(II), Fe(III) from sulfate solutions by Kelex 100 in xylene at three temperature levels. Kinetic experiments for the single metals and binary metal system simultaneously are planned. The composition space for these three cases are shown in Table 3. The Liquid Jet Recycle Reactor (LJRR) used for the experiments required minor modifications for the Cu(II) reaction. A one mm path length flow cuvette is needed for the Fe(III) system due to the high molar absorptivity of the iron complex. The kinetic experiments for simultaneous extraction of both metal ions will require development of an organic phase sampling technique and subsequent analysis of the solution for metals by an aqueous stripping and atomic absorption spectrophotometry. The analysis technique has been developed.

Experiments for the Cu(II) system were initiated in March, 1980. The kinetic data should be generated and analyzed

TABLE 3

COMPOSITION RANGE OF SPECIES FOR KINETIC STUDIES WITH LJRR

SYSTEM	MOLAR CONCENTRATION $\times 10^2$				
	$[\text{Fe}]_{\text{T}}$	$[\text{Cu}]_{\text{T}}$	$[\text{HR}]$	pH	EXPRS.
$\text{Cu}^{++}/\text{SO}_4^{=}/\text{Kelex 100/xylene}$	--	0.5, 1.0, 2.0	2.0, 4.0, 6.0	0.7 ± 0.2	26
$\text{Fe}^{+++}/\text{SO}_4^{=}/\text{Kelex 100/xylene}$	0.5, 1.0, 2.0	--	2.0, 4.0, 6.0	0.7 ± 0.2	26
$\text{Cu}^{++}/\text{Fe}^{+++}/\text{SO}_4^{=}$ Kelex 100/xylene	0.5, 1.0, 2.0	0.5, 1.0, 2.0	2.0, 4.0		16

by September 1, 1980.

(4) Single Stirred Tank Reactor Studies

Analysis

A model based on the film theory was proposed (2) to describe the flux of metal complex when multiple surface reactions occur and the time scale of the mass transfer process is of the same order as the surface chemical reaction process. These flux expressions will be combined with a two phase model for single and multiple staged continuous flow stirred tank reactor(s). The resulting expressions would permit calculation of the bulk compositions of the two metal complexes in the organic phase exiting the stirred reactor and thus prediction of extent of extraction and selectivity of the extraction process to Cu(II) over Fe(III). The model depends upon the nature of the intrinsic kinetic expressions for the rates of copper and iron extraction times in addition to the exchange reaction between the two complexes. Also, operating conditions and properties of the dispersion must be specified for calculations to be made.

Analysis of the above model was made assuming the reactions of the copper chelatin, iron chelation and liquid exchange reactions can be described by bimolecular second order kinetic expressions. The analysis, restricted to a single ideal stirred tank reactor, leads to two nonlinear algebraic expressions which can be solved for the concentrations of metal complexes in the effluent of the reactor. The equations are in terms of reactor operation variables, physical parameters

and rate coefficients. The model will be placed on the computer and extended to represent a battery of stirred reactor-settlers operated in co-current or counter current fashion. These models can readily accept the reaction kinetics models being generated experimentally. Predictions of conversion and selectivity can be made from the computer analysis.

Experimental

The 1 liter CSTR flow system to be used for the dispersed phase reactor studies has been modified. A new reactor was designed and the reactor heads were machined from 304 stainless steel to minimize difficulties in reactor operation. Additional features include drop generators with attendant continuous phase addition to prevent mass transfer during drop formation. A modified and remachined photomicrographic assembly will permit precise alignment of optical guide for illumination with micro photographic eyepiece. The AKUFVE separation centrifuge and spectrophotometer have been placed on stream. Preliminary drop size measurements indicate good data can be obtained during batch operation. Preliminary test and shake-down of the apparatus are scheduled for March and April, 1980.

Personal and P. I. Effort on Program

The personal employ during this period of funding includes Mr. E. Ho (M.S. Student, June 1, 1979), Dr. E. J. Wang (Special project, June 1, 1979), Mr. C. K. Lee (M. S. Student, January 1, 1980, and the principal investigator.

The principal investigator devoted 1 summer month in 1979 and 1.71 academic months from September, 1979 through

May 15, 1980. Total percentage effort throughout the year is 22.5%. 19% effort will be spent through May 30, 1980.

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- (2) L. L. Tavlarides, "Studies on Selective Metal Ion Extraction for Multiple Ion Exchange Reactions in Liquid-Liquid Systems," Grant DE-AS02-79ER10406, Office of Basic Energy Sciences.
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Figure 1. Extraction of Cu(II) Fe(III) in aqueous sulfate solution by 0.1M Kelex in xylene at 24°C; Δ - Cu(II) alone, 0.0315M; $\square$ - Fe(III) alone, 0.0362M; $\blacktriangle$ - Cu(II) at 0.0323M and $\blacksquare$ - Fe(III) at 0.0324M, mixture.

Figure 2. Extraction of Cu(II) and Fe(III) in aqueous sulfate solutions by 0.1M Kelex 100 in xylene at 24°C. ● - Cu(II) at 0.0354M and ■ - Fe(III) at 0.0344M (initial concentrations if completely dissociated).

Figure 3. Log D versus pH plot for chemical equilibrium studies of iron from sulfate solutions with 0.5M Kelex 100 in xylene.