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INTERFACIAL CHEMISTRY IN SOLVENT EXTRACTION SYSTEMS

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FINAL REPORT

The interfacial chemistry that occurs in the liquid/liquid extraction of metals ions still remains very incompletely understood at the molecular level. The objective of this comprehensive research program has been to further the fundamental understanding of this complex chemistry by systematically investigating the interfacial behavior of extraction reagents and their interactions with metal ions at both *macroscopic* (liquid/liquid) and *microscopic* (reversed micelles) interfaces. Although the importance of the macroscopic interface is well recognized, it is less appreciated that microscopic interfaces, i.e., association microstructures such as reversed micelles, are often present under practical conditions and play a key role in liquid/liquid extraction. An improved knowledge of the interfacial behavior of extractant molecules is of the utmost importance as it relates to the efficacy (extent, selectivity and rate) of the extraction process.

During the recent grant period we have more intensively investigated the physicochemical nature of metal-extractant aggregates (or microscopic interfaces) in the organic phase of acidic organophosphorus extraction systems from the perspective of colloid and surface science. Since industrial extraction systems are very complex, we emphasized the study of the aggregation behavior in model extraction systems of pure metal salts of bis(2-ethylhexyl)phosphoric acid (HDEHP) (e.g., NaDEHP, $Ni(DEHP)_2$, $Co(DEHP)_2$) or bis(2-ethylhexyl) sulfosuccinate, whose sodium salt (AOT) is the classical surfactant used often in studies of the structure and properties of reversed micelles, to eliminate any possible uncertainty in the metal-extractant complex composition. This approach evolved into a new initiative that utilized molecular modelling in order to clarify the molecular structure of metal-extractant micellar aggregates for which information is very difficult to obtain from direct experimental measurements. Significantly, we have obtained a number of novel findings that are contrary to conventional views of both the fundamentals of reversed micellar formation and growth as well as liquid/liquid extraction. These findings take on additional importance in view of the general increasing interest in micellar enhanced separation processes. In addition, we have continued to make significant progress in our efforts to develop new methodologies for characterization of the physicochemical nature of the macroscopic liquid/liquid interface by using advanced laser techniques.

The research productivity has been nothing short of excellent. This is especially so in view of the very difficult and challenging measurements that we proposed for investigating the structure and dynamics of the liquid/liquid interface, as well as our accomplishments in developing totally new concepts such as the "open water-channel" model of reversed micelles. Eighteen (18) papers and abstracts have been published since the submittal of our last three-year DOE progress report. Furthermore, several additional papers are in various stages of publication and preparation. In addition, we have given twelve (12) presentations describing various aspects of our liquid/liquid extraction and related research activities.

Association Microstructures in Liquid/Liquid Extraction

In previous studies of the liquid/liquid extraction of metal ions by HDEHP we reported that there appears to be three regimes of aggregation behavior and that the extent and rate of metal extraction are of practical importance when the metal-extractant complexes form association microstructures, namely, reversed micelles. Water molecules play an important role in the structure and function of reversed micelles. It is generally accepted that water is solubilized in the polar core of reversed micelles and that the size of reversed micelles increases with an increase in the amount of water present. Significantly, we have obtained a number of novel findings contrary to conventional view of reversed micelles.

Although the elucidation of the molecular mechanism associated with cobalt/nickel separation by HDEHP has been of significant interest, the knowledge of the aggregation behavior of its sodium salt (NaDEHP) in nonpolar organic solvents is also important. NaDEHP can also form in liquid/liquid extraction systems when the aqueous phase pH is adjusted with sodium hydroxide. Pure NaDEHP in *n*-heptane is the *simplest* model of an acidic organophosphorus extraction system one can study, and as such, this system was considered an excellent candidate for a base line study of metal-extractant aggregation behavior. Several very significant findings on the solution behavior of NaDEHP/*n*-heptane and NaDEHP/*n*-heptane/water systems have been made, although not every finding is noted in this report.

For example, NaDEHP in *n*-heptane was found to form *giant* quasi-one-dimensional or rodlike reversed micelles which violently contrasts with the literature view that the average micellar aggregation numbers in apolar (or nonpolar) media are much smaller than those in aqueous media. Furthermore, water was found to act as both an antimicellization agent and an antimicellar growth agent - behaviors which are markedly opposite to those for (AOT)/apolar medium systems. Also, the current literature views that the formation of reversed micelles in apolar media takes place over a wide range of surfactant concentration and that reversed micellization is a step-wise aggregation process appear to be, at least, not generally valid in nonaqueous surfactant solutions. As such, we presented a model that accounts for the effect of water on the size of the rodlike reversed micelles and suggests electrostatic interactions are the primary driving force for the growth of NaDEHP reversed micelles.

The solution behavior of the nickel (II) and cobalt (II) salts of HDEHP in *n*-heptane has also been investigated. The experimental findings are not consistent with the view that divalent transition metal-bis(2-ethylhexyl) phosphate complexes form polymers in nonpolar organic solvents. In fact, the concentration and temperature dependence of the size of the macromolecular species is typical of the solution behavior of surfactant association microstructures. We therefore proposed that the process for the formation of macromolecular species is an aggregation (or reversed micellization) of the $M(DEHP)_2$ complex monomers (two DEHP anions form a chelate with one divalent transition metal ion) through nonchemical or physical interaction forces to form rodlike reversed micelles rather than a polymerization via chemical or coordination bonds as believed by many authors in the literature.

In these earlier studies of the nickel (II) salt of HDEHP, the solubilized water was proposed to exist in the inner core of the cylindrical (or rodlike) reversed micelles consistent with the classical view of reversed micelles. However, upon careful examination, this interpretation is not fully consistent with what is known about the extent and selectivity of metal ion extraction by HDEHP. CPK space-filling molecular models show that $Ni(DEHP)_2.H_2O$ complexes form quasi-one-dimensional association microstructures or reversed micelles with two water channels. Thus, we recently hypothesized that the solubilized water molecules are present in "open" water channels that are in contact with the nonpolar solvent rather than exist in a "closed" water channel in the polar core of the rodlike micellar aggregates. This model is distinctly different than that of the conventional view of reversed micellar structures, wherein water is solubilized in the inner core of the reversed micelles. Nevertheless, the proposed "open water-channel" model for reversed micelles of $Ni(DEHP)_2.H_2O$ complexes is consistent with the known octahedral structure, the maximum W_o (mole ratio of water to metal-extractant complex) value and its temperature dependence.

The question of the location of the water molecules in the reversed micelles which form during the liquid/liquid extraction of nickel by HDEHP needed to be resolved, i.e., whether the water molecules exist inside (classical model) or outside (open water-channel model) the rodlike reversed micelles. Therefore, ^1H-NMR spectroscopy was used to investigate the nature of the water environment in a model system of $Ni(DEHP)_2$ reversed micelles to test the proposed open water-channel model. AOT was selected for comparison because, as indicated earlier, its structure is similar to that of HDEHP and its reversed micelles are known to solubilize water molecules in the polar core of the micellar aggregates. Since the unpaired electrons on the nickel atom of $Ni(DEHP)_2$ will create a magnetic field which opposes the applied field, thereby causing higher proton chemical shifts, $Ni(AOT)_2$ was also examined to account for the paramagnetic effect.

^1H-NMR spectra of $Ni(DEHP)_2$, AOT and $Ni(AOT)_2$ in both *n*-heptane and chloroform were obtained as a function of the water content. The chemical shifts for the $-CH_3$, $-CH_2$ and H_2O peaks are higher for $Ni(DEHP)_2$ reversed micelles than those for AOT and $Ni(AOT)_2$ reversed micelles. This indicates that the electronic environment around the different protons of the $Ni(DEHP)_2$ reversed micelles is affected directly and by reflection from other atoms by the field created by the unpaired electrons of the nickel atoms (paramagnetic effect) as well as by the location of the water molecules. The proton chemical shifts for $Ni(AOT)_2$ reversed micelles were similar to those for AOT reversed micelles where the water molecules are found inside the micellar core. The ^1H-NMR results indicate that the water molecules are likely located inside $Ni(AOT)_2$ reversed micelles in a manner similar to those in AOT reversed micelles, whereas the water molecules associated with the $Ni(DEHP)_2$ micelles seem to be situated outside in the channels between the hydrocarbon tails of the $Ni(DEHP)_2$ complexes, which is consistent with CPK molecular models, thereby providing initial evidence for the open water-channel model.

Molecular Modelling of Metal-Extractant Aggregates

Traditionally, metal-extractant complexes in the organic phase under low loading (dilute) conditions have been characterized by slope analysis. On the other hand, as the loading increases, the composition of the metal-extractant complexes appears to change and metal-extractant aggregates form. Unfortunately, relatively little knowledge is known about the physicochemical nature of the metal-extractant aggregates, described as either polymers or reversed micelles in the technical literature, for which information is very difficult to obtain from direct experimental measurements in highly loaded systems. One approach, however, which has great potential for revealing the molecular structure of metal-extractant aggregates is that of molecular modelling.

Molecular modelling techniques were employed to study the aggregation of metal salts of HDEHP in *n*-heptane to improve the understanding of the liquid/liquid extraction of nickel (II) and cobalt (II) ions. The nickel (II) salts of bis(2-ethylhexyl) phosphonic and phosphinic acid were also examined to provide more insight on structure-extractability-selectivity correlations. In particular, the organization of the metal-extractant complexes, the nature of the intermolecular forces, and the orientation and location of water molecules in the macromolecular metal-extractant species that form under conditions (concentration, solvation, etc.) simulating those of practical extraction were determined. Both geometrical optimization and molecular dynamics were employed using HyperChem and Sybyl molecular simulation software.

Molecular modelling showed that $Ni(DEHP)_2.H_2O$ complexes can exist as quasi-one-dimensional (rodlike) aggregates via hydrogen bond, van der Waals, and electrostatic forces. One of the most stable aggregates (based on energy minimization) consists of five complexes, which is in agreement with earlier VPO results that the mean aggregation number is about 5.2. Close examination of the optimized structure indicates that the octahedral $Ni(DEHP)_2.H_2O$ complexes are organized in a slip pattern with a mean distance of 0.54 nm between the nickel atoms. Furthermore, the coordinated water molecules form an organized network of intra- and intercomplex hydrogen bonds with the oxygen atoms of the oxyalkyl groups and the acidic oxygens of other complexes. On the basis of the hydrogen-bonding nature of the association of the $Ni(DEHP)_2.H_2O$ complexes, it can be concluded that the aggregates are reversed micelles. It is also evident that the (solubilized) water molecules are located in the two channels along the axis of the aggregate between the hydrocarbon tails of the complexes. This hydrogen-bonded network of water molecules bridges the complexes and provides an additional attractive force which reduces the distance between nickel atoms to 0.50 nm, thereby enhancing the stability of the reversed micellar aggregates. Although the nickel (II) salts of the phosphonic and phosphinic acid analogs also form rodlike reversed micelles, the surface of the micellar structures tends to be more lyophilic (or hydrophobic) in the order phosphoric < phosphonic < phosphinic because of structural differences associated with the amount of solubilized water which also decreases in the same order. Significantly, open water channels were not present in the surface of the rodlike reversed micelles of the cobalt (II) salt of HDEHP.

In conclusion, molecular modelling directly confirms the open water-channel model, where water molecules can be localized in compartments on the outside (surface) of rodlike reversed micelles. Furthermore, molecular simulation shows the dramatic effect that even minor changes in the extractant structure or metal ion substitution can have on the nanostructure of reversed micelles of acidic organophosphorus extraction reagents. Significantly, the molecular modelling study indicates that metal ion extractability and selectivity can be controlled by proper manipulation of the lyophobic/lyophilic character of reversed micellar structures, thereby illustrating the important role played by reversed micelles in the liquid/liquid extraction of metal ions. Realistic extraction models thus need to incorporate reversed micellar and other association microstructure effects.

Structure and Dynamics of Macroscopic Liquid/Liquid Interface

In the extraction of metal ions from an aqueous solution to a nonpolar organic diluent, the liquid/liquid interface plays a key role since the mass transport of ions occurs across the interface via an "interfacial reaction" between the metal ions and the extractant molecules. Although investigations in the past have provided valuable insight into the interfacial behavior of extraction reagents, it has become increasingly difficult to obtain new fundamental information from classical surface-chemical approaches. Significant advances in understanding the physicochemical principles underlying interfacial systems and processes can only be obtained if more sophisticated and recently developed techniques, such as advanced laser techniques, are applied.

The objective of this phase of the research program was to characterize the properties, structure and dynamics of the extractant-laden liquid/liquid interface of HDEHP/*n*-heptane/metal salt solution systems. In the past it has not been possible to investigate the macroscopic liquid/liquid interface in liquid/liquid extraction systems using fluorescence spectroscopic techniques due to the strong fluorescence contributions from the bulk (both organic and aqueous) phases, which are impossible to distinguish and separate from that of the interfacial region. We therefore are attempting to study a model interfacial film obtained by spreading quantitatively an organic phase containing both metal-extractant complexes and fluorescent probe molecules on an aqueous subphase. Laser-induced fluorescence measurements will then be performed using the pulsed Nd:YAG/dye laser system available in our laboratory to determine, for example, the fluorescence decay of a pyrene-labelled HDEHP analog. The fluorescence measurements should provide valuable information on the question of the formation of reversed micelles of metal-extractant complexes in the macroscopic liquid/liquid interface. Furthermore, we will measure the interfacial properties (dynamic interfacial elasticity and viscosity) as a function of the apparent thickness of the spread interfacial film. We expect that such an investigation will give important clues to understand the variation of the physicochemical properties in the interfacial region in real-life liquid/liquid extraction systems.

In this period we attempted to clarify the structure of adsorbed monolayers at the air/water interface prior to extending the advanced laser techniques to the liquid/liquid interface of model and practical extraction systems. In an earlier surface laser light-scattering (SLLS) study we obtained unique results of far-reaching implications regarding the interfacial structure of aqueous and apolar surfactant solutions. In the liquid/liquid extraction community it is generally assumed that the macroscopic liquid/liquid interface is homogeneous over the entire extractant concentration range. On the other hand, there are several reports in the general surfactant literature that a first-order phase change occurs (mainly from theoretical arguments) in the surface of dilute aqueous surfactant solutions. If so, then perhaps a similar transition occurs at liquid/liquid interfaces such as those of metal ion extraction systems.

SLLS measurements were performed on aqueous solutions of hexaethyleneglycol mono *n*-dodecyl ether ($C_{12}E_6$) since nonionic surfactants form large micellar aggregates in the bulk aqueous phase (and presumably large "surface aggregates"). The SLLS apparatus was realigned and upgraded with several new optical components. The high-frequency capillary wave experiments detected a relaxation process occurring in a relatively narrow $[C_{12}E_6]$ range where the measured dynamic interfacial elasticity was greater than the static interfacial elasticity. This non-agreement between dynamic and static interfacial elasticity values was previously reported by our laboratory, but it was considered likely due to an experimental artifact at that time. However, in view of its experimental duplication, the artifact explanation appears to be ruled out. Instead, the preferred conclusion is that the observed relaxation is consistent with a second-order or higher order transition occurring in the soluble monomolecular film. Independent confirmation of this significant finding by another experimental method would be most significant.

Steady-state and time-resolved fluorescence spectroscopy was therefore employed to study adsorbed $C_{12}E_6$ monolayers of aqueous solutions containing the fluorescent probe pyrene under controlled oxygen and humidity conditions. First, however, the Nd:YAG laser cavity had to be realigned by a Spectra Physics engineer during a service visit to Auburn University. In addition, the harmonic generator had to be shipped to the Spectra Physics service center for repair. The Nd:YAG laser, dye laser and wavelength extender were then realigned, and a preliminary experimental chamber suitable for the investigation of spread interfacial films was fabricated. Laser-induced fluorescence measurements showed that it is feasible to study the microviscosity and polarity of the probe microenvironment in adsorbed (or spread) interfacial films. However, the fluctuations in the Nd:YAG/dye laser intensity were too high, and therefore we were not able to determine if the fluctuations of the emitted fluorescence were due to a heterogeneous interfacial film on account of a "phase transition" or due to the probe laser beam itself. As such, a photodiode was purchased for use to monitor the intensity of the incident laser beam and, hence, permit the normalization of the measured fluorescence intensities. Unfortunately, the purchased photodiode was not able to measure the fluctuations in the laser intensity which seemed to be too low to be detected by the photodiode but high enough to cause important differences in the fluorescence spectra. Future research on the structure and dynamics of the macroscopic liquid/liquid interface will require improvements in the experimental setup for laser-induced fluorescence measurements.

Interfacial/Transport Phenomena Within Supported Liquid Membranes

It was the intent to initiate a novel study of the interfacial/transport phenomena that occur in the permeation of metal-extractant species, both in the form of metal-extract complexes and reversed micelles, through a supported liquid membrane (SLM). In our experimental approach to the problem, a state-of-the-art surface force apparatus (SFA) was to be employed. Furthermore, the SFA was to be incorporated into another laser setup that had been modified so ultra-violet FRAP (fluorescence recovery after photobleaching) measurements could be performed. The objective was twofold: (1) to directly measure "intrapore" diffusion in well-defined model pores between the two solid (silica) surfaces of the SFA and (2) to characterize the relationships among the molecular size of the metal-extractant species, separation distance (pore size), adsorption, molecule-surface (pore wall) interactions and diffusional behavior between silica surfaces chemically modified to mimic those of microporous membranes.

The design and fabrication of the MASIF (**M**easurement and **A**nalysis of **S**urface **I**nteractions and **F**orces) surface force apparatus and the prototype spectroscopy attachment were undertaken in cooperation with Australian Scientific Instruments (ASI), Canberra, Australia. The beta versions of the basic MASIF apparatus and the spectroscopy attachment were received at Auburn University in September 1995 and May 1966, respectively. However, since the MASIF arrived at Auburn University, it has experienced a number of mechanical and electronic problems. Two years of effort and two visits by ASI personnel in December 1996 and March 1997 have solved these initial problems that were caused in large part by the need to couple newer MASIF components with older counterparts. The hardware has been completely updated as well as the software upgraded. The basic MASIF apparatus is now fully operational with regard to surface force measurements. However, a fully working spectroscopy attachment remained elusive, but importantly, design modifications have been developed on the basis of experiments with the prototype attachment. Although the full potential of the proposed surface force/laser spectroscopy system is yet to be realized, the completion of a new spectroscopy attachment will permit the study of hindered diffusion of metal-extractant species as a function of the pore size in a model supported liquid membrane (SLM) system.

PERSONNEL

Principal Investigator

Professor Ronald D. Neuman was principal investigator for the DOE research grant.

Postdoctoral Associates

Dr. Parthasarathi Shah used steady-state and time-resolved (decay) fluorescence techniques in studies of model extractant films.

Dr. Taleb H. Ibrahim applied molecular simulation techniques to examine the physicochemical nature of metal-extractant species.

Graduate Students

Taleb H. Ibrahim employed ^{1}H-NMR, FTIR and molecular modelling techniques to investigate the nature of the water environment in Ni(DEHP)$_2$ reversed micelles.

Orlando J. Rojas utilized the surface laser light-scattering technique to measure the interfacial rheological properties of model extractant films.

DEGREES GRANTED

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2. Ibrahim, T.H., "Molecular Modelling of Reverse Micelles," PhD Dissertation, Auburn University, Auburn, AL, 1997.

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1. Neuman, R.D. and Ibrahim, T.H., "Novel Structural Model of Reversed Micelles: The Open Water-Channel Model," *Langmuir*, **15**, 10, 1999.

2. Yu, Z.J., Ibrahim, T.H. and Neuman, R.D., "Aggregation Behavior of Cobalt (II), Nickel (II) and Copper (II) Bis(2-Ethylhexyl)Phosphate Complexes in *n*-Heptane," *Solvent Extr. Ion Exch.*, **16**, 1437, 1998.

3. Wu, J., Zhou, N.F., Wang, D., Yu, Z.J., Neuman, R.D. and Xu, G., "Structure and Aggregation Behavior of Cobalt-Di(2-Ethylhexyl) Phosphoric Acid Complexes in Solvent Extraction Systems," *Science in China* (Series B, Engl. Edition), **41**(1), 36, 1998.

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TECHNICAL ABSTRACTS

1. Neuman, R.D., "Molecular Modelling of Reversed Micellar Agregates in Liquid/Liquid Extraction," Abstracts of Third DOE/BES Separations Research Workshop, Savannah, GA, May 1999.

2. Neuman, R.D. and Ibrahim, T.H., "Molecular Modelling of Reversed Micelles in Solvent Extraction Systems," Abstracts of Adsorption, Ion Exchange and Solvent Extraction Symposium, TMS Annual Meeting, San Antonio, TX, February 1998.

3. Ibrahim, T.H. and Neuman, R.D., "Molecular Modelling of Reverse Micelles," Abstracts of 71st Annual Colloid and Surface Science Symposium, University of Delaware, Newark, DE, June 1997.

4. Neuman, R.D., Yu, Z.J. and Ibrahim, T.H., "Microscopic Interfaces in Solvent Extraction," Abstracts of the 1995 International Chemical Congress of Pacific Basin Societies, Honolulu, Hawaii, December 1995.

5. Neuman, R.D., "Interfacial Chemistry in Solvent Extraction Systems," Abstracts of 2nd Biennial DOE/BES Separations Research Conference, Santa Fe, NM, June 1995.

6. Yu, Z.J. and Neuman, R.D., "Giant Rodlike Reversed Micelles and Bicontinuous Microemulsion in Sodium Bis(2-Ethylhexyl)Phosphate/*n*-Heptane/Water System," Abstracts of 68th Annual Colloid and Surface Science Symposium, Stanford University, Stanford, CA, June 1994.

7. Yu, Z.J. and Neuman, R.D., "Giant Rodlike Reversed Micelles and Sharp Transition Between Reversed Micelles and Microemulsions in Sodium Bis(2-Ethylhexyl)Phosphate/*n*-Heptane/Water System," Abstracts of 85th Annual American Oil Chemists' Society Meeting, Atlanta, GA, May 1994.

8. Kuo, C.H. and Neuman, R.D., "Surface Viscoelasticity of Surfactant Solutions Measured by Surface Laser Light Scattering," Abstracts of 85th Annual American Oil Chemists' Society Meeting, Atlanta, GA, May 1994.

PRESENTATIONS

1. Neuman, R.D., "Molecular Modelling of Reversed Micellar Aggregates in Liquid/Liquid Extraction," Invited presentation at Third DOE/BES Separations Research Workshop, Savannah, GA, May 1999.

2. Neuman, R.D. and Ibrahim, T.H., "Molecular Modelling of Reversed Micelles in Solvent Extraction Systems," Invited presentation at Adsorption, Ion Exchange and Solvent Extraction Symposium, TMS Annual Meeting, San Antonio, TX, February 1998.

3. Neuman, R.D., "Molecular Modelling of Metal-Extractant Aggregates in Liquid/Liquid Extraction," Invited presentation at Argonne National Laboratory, Argonne, IL, November 1997.

4. Ibrahim, T.H. and Neuman, R.D., "Molecular Modelling of Reverse Micelles," Paper presented at 71st Annual Colloid and Surface Science Symposium, University of Delaware, Newark, DE, June 1997.

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