

**D103: VARIATION OF PROPERTIES OF AQUEOUS PHASE IN SOLVENT SUBLIMATION FOR BUTYL ACETATE RECOVERY FROM PENICILLIN WASTEWATER AND SYNTHETIC SOLUTION**Xinghua Sun<sup>\*\*\*</sup>, Zhidong Chang<sup>\*</sup>, Mingmei Wang<sup>\*</sup>, Shufeng Shen<sup>\*\*\*</sup>, Xin Hu<sup>\*\*\*</sup>, Zhi Xie<sup>\*</sup> and Huizhou Liu<sup>\*</sup><sup>\*</sup> Laboratory of Separation Science and Engineering, State Key Laboratory of Biochemical Engineering, Institute of Process Engineering, Chinese Academy of Sciences, Beijing, China<sup>\*\*\*</sup> Graduate School of the Chinese Academy of Sciences, Beijing, China

Solvent sublimation is effective to the recovery of butyl acetate (BA) from the wastewater of penicillin plant, but its efficiency in synthetic solution is much less. Experiment results on the variation of properties of the aqueous phase showed that the surface tension and electrical conductivity of the wastewater increased during the sublimation. However surface tension of synthetic solution first increased, then decreased, and increased again or decreased a little during the sublimation. Viscosity of the wastewater also decreased during the sublimation. Chemical oxygen demand (COD) of the wastewater decreased after the sublimation, and such variation compared with that in conventional azeotropic distillation (stripping) was discussed. It shows that the properties of wastewater are different from the synthetic solution because of various biological materials, surfactants and polymers in the wastewater.

**D105: SEPARATION OF METALS FROM SPENT PORTABLE BATTERIES BY LIQUID-LIQUID EXTRACTION WITH CYANEX 272**

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A hydrometallurgical route consisting of classification, dismantling, sulphuric acid leaching and liquid-liquid extraction steps has been proposed to recover metals from spent portable batteries. In this work, the liquid-liquid extraction step with Cyanex 272 is highlighted in order to separate Zn, Mn, Ni, Cu, Al, Cd, Li and Co which were found as main metal content of NiCd, NiMH and lithium batteries. Equilibrium tests were carried out by contacting a synthetic aqueous solution containing 0.1 mol/L of each metal with an organic solution of 0.3 mol/L Cyanex 272 dissolved in commercial kerosene (Escaid 110) at 50°C. Analysis of metal extraction at different pH values have shown that  $pH_{1/2} = 2.5-3.0$  for Zn and Al,  $pH_{1/2} = 4.0-4.5$  for Mn, Cd, Cu and Co,  $pH_{1/2} = 6.5$  for Ni and  $pH_{1/2} = 8.0$  for Li. Data fitting to an equilibrium model proposed to the test system by the EFChE (European Federation of Chemical Engineering) have indicated that  $MR_2(RH)_2$  and  $MR_2$  complexes (where M = Zn, Mn, Co, Cd and Cu) with Cyanex 272 co-exist in the organic phase depending on loading conditions.

**D106: IONIC LIQUIDS AS EXTRACTANTS OF PHENOLIC COMPOUNDS AND METAL IONS: INFLUENCE OF THE TYPE OF IONIC LIQUID**

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This work presents some results concerning the use of 1-(n-alkyl)-3-methylimidazolium-(C<sub>n</sub>mim), 1-(2-ethoxyethyl)-3-methylimidazolium-(C<sub>2</sub>OC<sub>2</sub>mim), and 1,3-bis(2-ethoxyethyl)-imidazolium-derived [(C<sub>2</sub>OC<sub>2</sub>)<sub>2</sub>im] ionic liquids (IL) in liquid-liquid separation processes. For this purpose, a series of methylimidazolium and imidazolium tetrafluoroborate and hexafluorophosphate ILs were prepared by standard synthetic methods and characterized by <sup>1</sup>H NMR. The density and viscosity of the water-equilibrated ILs were also determined. Equilibrium studies on the extraction of copper and zinc ions (300 mg/L) from chloride solutions and of some phenolic compounds (tyrosol and *p*-hydroxybenzoic acid) from aqueous media were performed. High extraction efficiencies of both metal ions were attained with BF<sub>4</sub> ILs, while the corresponding values with PF<sub>6</sub> ILs were in some instances about 50% lower. Concerning the extraction of the organic compounds it was found that the BF<sub>4</sub> ILs are also more efficient than the corresponding PF<sub>6</sub> derivatives. Furthermore, for the PF<sub>6</sub> ILs, the substitution of the alkyl group by an ether in the methylimidazolium structure resulted in increases of about 10% in the extractions of *p*-hydroxybenzoic acid and tyrosol. Finally, the ILs containing BF<sub>4</sub> were the most efficient to extract zinc (extraction > 90%). In addition, (C<sub>2</sub>OC<sub>2</sub>)<sub>2</sub>imPF<sub>6</sub> was also capable of removing more than 80% of the zinc from a 4.5 M NaCl solution. The same IL, (C<sub>2</sub>OC<sub>2</sub>)<sub>2</sub>imPF<sub>6</sub>, extracted 82% of the copper from a 4.5 M NaCl solution, while a value of 44% and 21% was obtained with the BF<sub>4</sub> ILs and with C<sub>4</sub>mimPF<sub>6</sub>, respectively.

### **D107: METHYL TERT-PENTYL ETHER (TAME) – A NEW SOLVENT FOR PHENOL EXTRACTION FROM WASTEWATER**

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Methyl tert-pentyl ether [1, 2, 3] is a new solvent, usable for the phenol extraction from the wastewater. Liquid-liquid experimental data in the system water-phenol-TAME show a much higher distribution coefficient for this solvent than for other solvents. The liquid-liquid equilibrium data, obtained with low prices in an extremely simple laboratory installation, have allowed the design of an industrial installation for phenol recovery from the wastewater in an industrial plant of a Romanian phenol producer. The process has been initially tested using a laboratory extraction column, provided with different packings.

Along with the liquid - liquid extraction column, the suggested installation is endowed with a solvent recovering system from (1) the extract phase, respectively TAME-phenol mixture, and (2) refined phase, respectively water-TAME mixture.

Because the solubility of TAME in water is lower than the one of the other suggested solvents (methyl *t*-butyl ether - MTBE), its recovery from the refined phase is easier.

The phenol extraction from acid water is another usage direction for proving the possibilities of also using it in this field of activity.

### **D108: SELECTIVE RECOVERY OF PHENOLS FROM PHENOLIC RESIN PLANT EFFLUENT BY LIQUID-LIQUID EXTRACTION**

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In this work the recovery of phenols from a phenolic resin plant effluent by liquid-liquid extraction has been studied. Several extractants were tested. The selected organic phase allowed a selective recovery of phenols towards formaldehyde by this technology. Concentration of phenols from 46 g/L in the phenolic resin wastewater to 28 wt. % was obtained. The stripping concentrate can be reused for phenolic resin production.

### **D109: RECOVERY OF ZINC FROM SULPHATE AQUEOUS SOLUTIONS USING HOLLOW FIBRE CONTACTORS**

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This work aims to study the extraction of zinc from sulphate aqueous solutions using a hydrophobic propylene membrane contactor. The organophosphorus compounds bis(2-ethylhexyl)phosphoric acid (D2EHPPA) and bis(2-ethylhexyl)thiophosphoric acid (D2EHTPA) were used as extractants. The influence of the initial pH of the feed phase on the overall mass transfer coefficient was determined for both systems. The effect of the aqueous and organic throughputs on the overall mass transfer coefficient was also analysed. Integration of the extraction and stripping stages was carried out using different volume phase ratios. Concentration ratios of about 18-fold with 90% recovery were obtained. The results were finally compared with the ones obtained by applying the Emulsion Liquid Membranes separation technique.

### **D111: STUDY OF PARAMETERS IMPORTANT IN THE PROCESS OF BIOSORPTION TO THE REMOVAL OF HEAVY METALS**

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The increase of the concern with the ambient question and of the severity of the legislation in the discarding of industrial and domestic containing metals has motivated the inquiry of new technologies for the recovery and metal removal. The removal of you put heavy for microbial biomass has been investigated as an alternative to the traditional methods. Thus it was studied removal of metals has copper and iron of discarded lubricating oil having used as biosorbent the seaweed *Sargassum* sp.

### **D112: SEPARATION PROPIONIC ACID/ACETIC ACID WITH BASIC POLYMERIC SORBENTS**

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Propionic acid is an important carboxylic acid applied in pharmaceutical, pesticide and foodstuff. During recovery of product propionic acid from the fermentation broths, it is important to maximize the selectivity for the desired acid to the additive acetic acid. In this work, three types of weakly basic resins, D301R, D301G, D392 and one neutral phosphine resin, TBP resin, were selected as the adsorbents, the uptakes of single component of propionic acid and acetic acid, as well as competitive uptakes of propionic acid and acetic acid have been measured. The dependence of pH values on the adsorption equilibrium was studied also. The results showed that Langmuir equation could be used to describe the sorption isotherms for both single carboxylic component (propionic acid and acetic acid) and two components of propionic and acetic acid. Despite of low uptake capacity, TBP resin gives a higher selectivity in the pH 3~5 range than D301R, D301G and D392.

### **D201: IONIC LIQUIDS FOR BTEX SEPARATIONS**

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Aromatic hydrocarbons (benzene, toluene, ethylbenzene and xylenes, BTEX) and aliphatic hydrocarbons are normally separated by liquid extraction, extractive distillation or azeotropic distillation. Separation of aromatics by extraction with ionic liquids would require fewer process steps and less energy consumption than extraction with conventional solvents, because ionic liquids have a negligible vapour pressure. The toluene/heptane selectivities at 10% toluene in the feed at  $T = 40^{\circ}\text{C}$  and  $75^{\circ}\text{C}$  with several ionic liquids are a factor of 1.5-2.5 higher compared to those obtained with sulfolane, which is a conventional solvent for the extraction of aromatic hydrocarbons from a mixed aromatic/aliphatic hydrocarbon stream.

### **D202: MIXED SOLVENTS AS ENVIRONMENTALLY BENIGN REPLACEMENT FOR BENZENE IN CAPROLACTAM EXTRACTION**

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Because of the strict legislation for currently applied solvents in the industrial extraction of caprolactam, being benzene, toluene and chlorinated hydrocarbons, a need exists for alternative, environmentally benign solvents. As replacement solvent mixtures containing a polar solvent and an alkane were proposed, in order to combine the favourable and eliminate the negative properties of both solvent types. In a screening procedure it was found that the caprolactam distribution ratio increased with increasing solvent (mixture) polarity, characterized by a more polar active group (ether, ester, ketone and alcohol), decreasing carbon chain length ( $\text{C}_{12}\text{-C}_6$ ) or increasing polar solvent fraction in the mixture. Based on a high capacity and a low mutual solvent solubility the mixture methylcyclohexane-heptanol (40 mass %) was selected as replacement solvent. Calculating the solvent to feed ratio and NTS needed to reach the required raffinate concentrations for the industrial DSM extraction process lay-out, it was found that in the forward extraction  $S/F = 3.0$  for both the mixed solvent and benzene, whereas  $\text{NTS} = 5$  and  $10$ , respectively. In the back-extraction  $S/F = 0.75$  and  $0.50$  and the  $\text{NTS} = 10$  and  $6$  for the mixed solvent and benzene, respectively.

### **D204: EXTRACTION OF SELECTED DYES WITH ALKYLENE CARBONATES**

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Propylene and 1,2-butylene carbonates enable very effective extraction of Methylene Blue. The use of a more hydrophobic 1,2-butylene carbonate is preferred for practical application. Separation is enhanced by addition of an electrolyte. The recovery of Methylene Blue with propylene and 1,2-butylene carbonates is significantly better, compared with the cloud point technique. Alkylene carbonates are not appropriate extractants for recovery of very hydrophilic Direct Yellow.

### **D205: EXTRACTION OF ZINC(II) AND IRON(III) FROM SPENT PICKLING SOLUTIONS OF HIGH ZINC(II) CONTENT**

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Zinc(II) or iron(III) extraction with tributyl phosphate (TBP) from spent pickling solutions containing high zinc(II) concentration is studied. Two technological approaches for the separation are considered: the selective extraction of iron(III) over zinc(II) with small portions of TBP, and zinc(II) selective extraction with an excess of TBP after iron(III) reduction to iron(II). None of the both versions enables selective extraction of iron(III) or zinc(II). The total removal of iron(III) needs several successive extractions with fresh TBP portions. Zinc(II) can be quantitatively recovered in the presence of iron(II) using 5-10 volume excess of TBP.

### **D206: CLOUD POINT SEPARATION OF PHENOLS**

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The removal of phenols from aqueous streams by the cloud point extraction technique was studied. New oxyethylated methyl dodecanoates with various average degrees of oxyethylation (n) were used as nonionic surfactants. The effect of surfactant hydrophobicity, electrolyte content, the hydrophobicity of solutes on recovery of phenols was determined. The overheating of 20°C over CP was selected as sufficient. It was found that the recovery of phenols depended mainly on the hydrophobicity and proton donor acidity of solutes. The recovery of phenols was enhanced by the electrolyte presence. The obtained results could be explained by the linear solvation energy relationship (LSER) proposed by Abraham for micellar systems.

### **D207: SEPARATION OF PHENOLS BY MICELLAR ENHANCED ULTRAFILTRATION**

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Micellar enhanced ultrafiltration (MEUF) of solutions containing phenols was studied. Oxyethylated fatty acid methyl ester (OMD), alkylpolyglucoside (APG), sodium dodecylsulfate (SDS) and hexadecyltrimethylammonium bromide (CTAB) were used as representatives of nonionic, anionic, cationic surfactants. It was found that MEUF enable high recovery of phenols without using any hydrocarbon solvents. CTAB and SDS are appropriate surfactants for this technique. The recovery of phenols could be estimated by the micelle binding constants or from linear solvation free-energy relationships according to the Abraham model.

### **D208: THE USE OF IONIC LIQUIDS IN SOLVENT EXTRACTION PROCESSES**

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In this study, room temperature ionic liquids (RTILs) have been investigated as an alternative to volatile organic solvents (VOSs), in solvent extraction processes. The RTILs have been used in a range of applications by various researchers due to their very low vapour pressure, ability to dissolve a range of organic, inorganic and organometallic compounds, and their immiscibility with water and ability to form biphasic systems. This research work has focussed on determining the physical properties of two ionic liquids, namely 1-butyl-3-methylimidazolium hexafluorophosphate [bmim][PF<sub>6</sub>] and 1-hexyl-3-methylimidazolium bis((trifluoromethyl)sulfonyl)imide [hmim][Tf<sub>2</sub>N] and comparing these properties to the conventional organic solvents, xylene/benzyl alcohol (BA) and xylene/tributylphosphate (TBP) to determine their potential as replacements solvents. Physical properties such as the interfacial tension, viscosity and density were determined. Also, the solubility of water in these solvents was measured. Distribution ratio versus pH were obtained for four systems [bmim][PF<sub>6</sub>]/water, [hmim][Tf<sub>2</sub>N]/water, xylene/TBP/water and xylene/BA using amine containing compounds as solutes.

**D209: A CONTINUOUS SOLVENT EXTRACTION PROCESS FOR THE VALORIZATION OF ZINC FROM SLUDGE LEACHING LIQUORS**

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CYANEX 301 dissolved in n-dodecan and modified by octanol-1 was used to extract zinc from a strong acidic sulphate ferrite's leaching liquor. From thermodynamics and kinetics studies, conditions for a selective and quantitative extraction were found. Cementations onto the leaching liquor prior to solvent extraction proved to be useful for both environmental sight and effective long term process running. Zinc was quantitatively stripped from its extracted organic complex using aqueous NaOH. Laboratory scale mixer-settlers tests achieved the required level of zinc separation within only two single extraction stages, confirming Mc Cabe & Thiele predictions. The global hydrometallurgical process flowsheet developed represents an adding value to ferritic zinc resources with an environmental clean option.

**D210: APPLICATION OF BLOCK COPOLYMER IN THREE-LIQUID-PHASE EXTRACTION SYSTEM**

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A novel three-liquid-phase extraction system (TES) composed of butyl acetate (BA), block copolymer [polyethylene oxide-polypropylene oxide-polyethylene oxide (PEO-PPO-PEO)] and ammonia sulphate aqueous solution  $[(\text{NH}_4)_2\text{SO}_4]$  as top, middle and bottom phase respectively has been developed. The copolymer recycling and partitioning behaviour of penicillin V has been studied in this system. Results showed that the copolymer could be purified and recycled and penicillin V of the filtrated ferment broth could be partitioned unevenly among the phases and purified in the top phase of this TES. About 90% (weight percent) of penicillin V could be distributed into the top phase around pH2.5 and only less than 0.1% left in the bottom phase.

**D211: EXTRACTION BEHAVIOR AND MECHANISM OF ORGANIC SOLUTES FROM DILUTE SOLUTION BY GREEN SOLVENT DIMETHYL CARBONATE**

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Dimethyl carbonate(DMC) was selected as green extractant to prevent pollution existed in current liquid-liquid extraction process. An experimental investigation of the distribution of organic solutes between DMC and water was carried out and the extraction mechanism of it was studied in this work. Distribution coefficients of 13 substances between DMC and water were determined by experiments. Also comparisons of the distribution coefficients with substance structures were made. Vanillin was selected as an example solute to study the extraction behavior of DMC in detail. Molecular structure of DMC and the interaction of DMC with other molecules were analyzed. Solute distribution coefficient between Octanol and water( $P$ ) was proposed as an auxiliary parameter to study extraction mechanics of DMC. Correlation results of the logarithmic form of distribution coefficients ( $\log D$ ) with hydrophobic parameter ( $\log P$ ) showed that they had a quite good linear relationship. This provided one good guide for the application of DMC as a green extractant.

**D301: BATH LIFE EXTENSION DURING ELECTROLESS NICKEL PLATING BY SOLVENT EXTRACTION**

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For the electroless nickel plating onto aluminum alloys, nickel preplating (strike plating) is necessary after zincate treatment to improve the adhesion of the nickel deposit. However, the life of the strike plating bath is short due to the accumulation of zinc in the bath. We have applied solvent extraction using acidic organophosphorous extractants, such as PC88A and Cyanex272, in order to extend the bath life by selectively removing zinc ion from the baths. This process has been in actual operation at a plating plant using a mixer-settler extractor for more than one year. Since the introduction of this process, the bath life has been extended 5-7 times longer when compared to the standard process.

### **D302: SOLVENT EXTRACTION AND SEPARATION OF PALLADIUM FROM RECYCLED AUTOCATALYSTS WITH A NEW-BRANCHED DIALKYL SULPHIDE**

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The experiment conditions of solvent extraction and separation of palladium from recycled autocatalyst with DIAS have been studied. The results shown that the extraction percentage in four cross-current extraction stages and stripping percentage in three counter-current stripping stages of palladium is 99.95%, 99.9% respectively. The recovery and purity of palladium are all 99%. The results indicated that the method has many advantages including high kinetics, continuous process, wide acidity range, high selectivity and recovery, short work flow, friendship work environment, and so on.

### **D303: EXTRACTION OF THE *CALENDULA OFFICINALIS* L. ESSENTIAL OIL AND IT'S CHEMICAL COMPOSITION**

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The use of the vegetable raw material as medicinal herbs has been increased in national and international companies. The oil of *Calendula officinalis* stimulates the granulocytosis and the fagocytosis helping the fight against the infections. The extraction of the oil was made through the adapted method that use water vapor. The identification of the compound was done by comparing the mass spectrums obtained with the ones of the data bank and by the Kovats index of each component. The results show that the lower drying temperature results in more oil extraction of this species, with the preservation of the monoterpenes.

### **D304: OPTIMIZATION OF INULIN EXTRACTION FROM CHICORY ROOTS**

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Chicory root is the most important tuberous root for industrial production of inulin which has a high fructose content. Inulin is a stored carbohydrate which can be found in more than 30,000 vegetable products. It is a fructooligosaccharide that is not digestible but is selectively utilized by bifidobacteria in the large intestine, making inulin-type fructans the prototype prebiotic. Recently, inulin was identified as an ingredient that substitutes fat or sugar. In this work, inulin samples were extracted from chicory roots by a hot water diffusion process. The methodology consisted of the election of two levels of bath temperature and water:root ratio (water mass/root dry mass) and their combinations in a experimental design to execute the experiments. Second order polynomial model, response surface and contour plot were generated and allow the choice of temperature and water:root ratio that gives the extraction condition with highest inulin concentration.

### **D308: EXTRACTION OF SUCCINIC ACID WITH TRIALKYLAMINE**

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The extraction of succinic acid from aqueous solution by trialkylamine dissolved in binary mixed solvent of n-octanol and toluene was studied. In addition to the influence of single factor on extraction, equilibrium experiments were designed by orthogonal experiment. By the treatment of the orthogonal experimental data, the main influence factor was obtained. Using mass action law, the liquid-liquid equilibrium model of complex extraction of polybasic acid has been induced, and physical extraction was also considered in the model. Comparing experimental data with model value, it could be held that the regression precision of model was high. IR spectrum was performed for information on the complexes and extraction mechanism, which confirmed the concurrence of hydrogen-bond and ionic-bond between succinic acid and amine. Combined the complex constant value of model with the spectrum, four kinds of complex compounds synthesized by succinic and trialkylamine were determined.

**D309: RECOVERY OF ZINC FROM GALVANIZED STEEL SCRAP USING HOT CAUSTIC LEACHING FOLLOWED BY SOLVENT EXTRACTION AND PRECIPITATION****G. V. K. Puvvada, R. Sridhar and V. I. Lakshmanan**

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In US approximately 60% of the zinc metal produced is consumed in galvanizing the steel. During galvanizing a thin layer of zinc coating on the steel surface is formed in order to protect steel from corrosion. The scrap that is generated from the galvanized steel materials is collected and recycled in steel mills to recover the iron value. During this process of recycling of scrap galvanized steel, the zinc is lost by forming a dust called the steel mill dust or EAF dust, which is classified as hazardous and therefore is being discarded in secured landfill. Several attempts were made by various investigators to recover the zinc value from the steel mill dust, however no suitable technology was found viable to date.

Process Research ORTECH Inc (PRO) has developed a new sustainable process for the recovery of zinc from galvanized steel scrap. The process consists of treating the zinc containing scrap in an alkaline solution to remove the zinc to produce a steel scrap free of zinc for recycle (Black Scrap) and treat the zinc containing solution by a novel solvent extraction process to recover value added zinc oxide as the product. The caustic is simultaneously recovered and recycled for leaching. This process reduces the environmental problem caused by the production of hazardous zinc containing fumes by the steel plants. This process not only avoids zinc containing land fill close to 0.5 million tones as EAF dust per year in North America, but also avoids emission of approximately 52 tones of CO<sub>2</sub> per tone of zinc produced from the mined ore.

**D311: STUDY ON LIQUID-SOLID EXTRACTION FLOTATION SEPARATION OF MERCURY (II) BY THE SYSTEM OF SODIUM CHLORIDE-AMMONIUM THIOCYANATE-CETYLPYRINIUM BROMIDE-WATER****Xinrong Wen and Changqing Tu**

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The liquid-solid extraction flotation separation of mercury (II) from other metal ions using the system of sodium chloride-ammonium thiocyanate-cetyl pyrinium bromide-water was studied. The results showed that with the amount of 1.0g NaCl and 0.5mL of a solution of 0.1mol/L NH<sub>4</sub>SCN and 1.0mL of a solution of 1.0×10<sup>-2</sup>mol/L cetylpyrinium bromide(CPB), Hg<sup>2+</sup> could be extraction floated quantitatively, and Cd<sup>2+</sup>, Ni<sup>2+</sup>, Mn<sup>2+</sup>, Co<sup>2+</sup>, Fe<sup>2+</sup>, Al<sup>3+</sup> could not be extraction floated, so Hg<sup>2+</sup> could be separated successfully from those metal ions by extraction flotation at pH5.0. This method has been used for the extraction flotation separation of Hg<sup>2+</sup> in the sample of synthetic water and the results were satisfactory.