

PROGRESS IN SCIENCE
AND TECHNOLOGY OF THE
RARE EARTHS

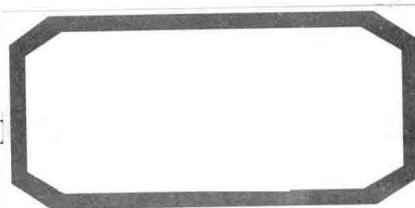
稀土科学与技术进展

中国稀土学会 编



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Progress in Science and Technology of the Rare



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内 容 简 介

本书内容主要为第八届国际稀土开发与应用研讨会的部分论文和摘要。国际稀土会议在中国、美国和欧洲国家轮流召开。迄今,中国稀土学会已成功举办八届国际稀土开发与应用研讨会(ICRE)。本书内容丰富,包括资源和环境领域的稀土地质矿山、采矿选矿、分离冶炼、循环利用和环境保护,以及稀土物理化学和稀土应用领域的永磁、发光、催化、储氢、超导、玻璃陶瓷、纳米材料等,反映了相关领域的最新科研动态。

本书可为科研机构、大专院校、政府部门、稀土企业等从事稀土采选冶、应用、环保工作的人员以及科研人员提供参考,也可供对稀土科研和产业感兴趣的有关人士阅读。

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Contents

Section A: Full Paper

A High-Efficient System of the Ionic-Liquid Containing CHON-Type Extractant for Extraction and Recovery of Rare-earth Resources	3
The Principle and Advantages of Fractional Extraction System with Two Inlets for Separating Rare Earths from Two Kinds of Material Liquid	9
Effects of Composite Powders of Cerium Oxide / Tourmaline on Diesel Combustion	17
Molecular Modeling and the Structures of the Lanthanide Complexes with Cyclopyridines, a Novel Class of Ligands I: Cyclotripyridine and Its Ln(III), Sc(III) and Y(III) Complexes	28
Study on Criticality Assessment of Rare Earths	36
Gradient Distribution of Dy in Diffusion Treated Nd-Fe-B Sintered Magnet	42
Effect of Minor Yttrium on Mechanical Properties and Microstructure of Die-Casting Magnesium Alloy AZ91D	50
Review on Application Technology of Light Rare Earth Elements La and Ce	59
Study on Sol-Gel-Foaming Method for Preparing a Novel Porous Ceria	66
Beneficiation-Hydrometallurgy Combination to Process a Phosphorus Rare Earth Ore	71
Broad-Band Absorption and Sensitized Fluorescence of $\text{Sr}_8\text{MgY}(\text{PO}_4)_7$: Tb^{3+} Phosphors by Codoping with Ce^{3+} Ions	84
Effect of the Sintering Behavior and Far Infrared Radiation Property of Rare Earth-Doped Iron Ore Tailings Ceramic	95
Study on the Change of Soil Erosion Pattern Using Rare Earths Elements Tracking Method	104
Lattice Boltzmann Simulation for the Process of Mass Transfer of Leaching Rare Earth ..	113

Section B: Abstract

Ceria: A Versatile Oxide for Energy and Environmental Applications	131
China's Rare-Earth Permanent Magnet Industry	132
C-Type and Fluorite-Type Rare Earth Oxides: Application for Functional Catalysts	133
Trends in Global Downstream Application and Forecasted Demand for Rare Earths in 2020	134
Rare Earth Phosphors for Lighting and Display	135
Status of Rare Earth Hydrogen Storage Materials and Ni-MH Batteries and Future Prospects	136

Application of Rare Earth Ions in Fast and High Density Scintillators	137
Crystallization Study of Functional Rare Earth Inorganic Materials	139
Rare Earth Oxide Materials: Exploiting Size, Spins and Spectra	140
Structure and Negative Thermal Expansion in RE Compounds	142
Design, Syntheses and Characterizations of Several Novel Long Persistent Phosphors	143
Molecular Modeling and the Structures of the Lanthanide Complexes with Cyclopyridines, a Novel Class of Ligands I: Cyclotripyrindine and Its Lanthanide(III) Complexes	144
REE Recycling by Advanced Magnetic Separation Nanotechnology	145
New Technique for Bayan Obo Mixed RE Concentrate Based on Valency Change and Complexation of Cerium with Fluorine and Phosphorus	146
Synthesis and Reactivity Studies of Rare Earth Polydentate Aryloxide Complexes	147
Design and Investigation of New Mold Fluxes for Heat Resistant Steel Containing Rare Earth Continuous Casting	149
Organometallic Rare Earth <i>N</i> -Heterocyclic Carbene Complexes for C—H Bond Activation and Catalysis	151
Recovery of Rare Earth Elements (REEs) from Pregnant Leach Solution of Apatite through Precipitation	152
Extraction and Separation of Ce(IV), F(I) and Th(IV) from Sulphuric Acid Leaching of Bastnaesite by a Novel Extractant Cextrant230	153
Recovery of Rare Earth From Leaching Solution of Ion-Adsorption Rare Earth Ores by Centrifugal Extraction with HEH(EHP) and D2EHPA	154
A Process of Rare Earths Leaching and Extraction Separation in Sulfuric Acid System with Magnesium Bicarbonate Solution, as Well as Magnesium Salt Wastewater Recycling	155
Recovery of Terbium and Gallium from TGG Crystal	156
Geochemistry of Rare Earth Elements within Jamaican Bauxite Deposits	157
The Effect of Rare Earth (Cerium) Ions Location on REY Zeolite Catalytic Activity and Product Distribution in FCC Process	158
Beneficiation and Leaching of Phosphate Process Streams for REE Recovery	160
Complex Processing of Phosphor Production Slag with Rare Earth Metals Recovery and Deriving of Silicon-Containing Solution	161
Novel Algorithm for Solving Minimum Extraction Amount in Multi-component Extraction System	163
Synthesis, Luminescence Properties and Mechanisms of Novel Long-Persistent Phosphorescence Materials $\text{Na}_2\text{CaSn}_2\text{Ge}_3\text{O}_{12}$, Ca_2SnO_4 and CaSnO_3 Doped by Rare Earth Ions	164
Beneficiation-Hydrometallurgy Combination to Process a Phosphorus Rare Earth Ore	166

Experimental Study and Thermodynamic Calculation of $\text{Lu}_2\text{O}_3\text{-SiO}_2$ Binary System	167
Promising Hydrogen Storage Properties of La-Y-Mg-Ca-Ni AB_3 -Type Alloys	168
Ionic Conductor Titanate Red Phosphor in LEDs and FEDs: DFT Calculation, Luminescent Characteristic, Thermal Stability and Conductivity Analysis	169
From Supramolecular Assembly to Luminescent Detection of Biomarkers	170
Optical and Scintillation Performance of Ce Doped $\text{Li}_6\text{RE}(\text{BO}_3)_3$ (RE=Gd, Y, Lu) Crystals for Neutron Detection	172
Control of Agglomeration During the Preparation of Nanometer Yttrium Oxide by Precipitation Method	173
Permanent Magnetic Nanomaterials beyond Neodymium-Iron Magnets	174
Single Crystal Growth and Performance of $(\text{Gd}_{0.9}\text{Lu}_{0.1})_2\text{Si}_2\text{O}_7\text{:Ce}$ Scintillator	175
Computational and Experimental Investigation of Scandia-Doped Zirconia Used in SOFCs	176
Chemical Control of Crystal Structure and Photoluminescence in Oxonitridosilicate Phosphors	177
The Synthesis and Luminescent Properties of Ce^{3+} -Activated Garnet Phosphor	178
The Effect of Gd on a Red Shift in Ce-Doped $\text{Y}_2\text{Si}_4\text{N}_6\text{C}$ UV-Excitation Phosphors for White LEDs	179
Auto-thermal Reforming of Ethanol into Hydrogen over Ni-La/ CeO_2 Catalyst	180
Rare Earth Ce-Based DeNO_x Catalysis Technology and Its Application	181
Effect of RE Element Pr on the $\text{CeO}_2/\text{Al}_2\text{O}_3$ Catalyst for Selective Catalytic Reduction of NO by NH_3	183
Rear Earth Doped NaYF_4 Microrod with Unusual Upconversion Luminescence Emission ..	184
Synthesis and Luminescence Properties of Novel Rare Earth-Doped Rare Earth Thiosilicate and Halothiosilicate for White LEDs	185
Application of Rare Earths in Thermal Barrier Materials	186
Structure Analysis and Properties of Eu^{3+} Doped Zinc Bismuth Borate Glasses Applied in YAG Phosphor-in-Glass for WLED	187
Preparation and Performance of Cerium Dioxide Film by Anodization and Heat Treating ..	188
A Dual-Emission Nanoarchitecture of Lanthanide Complex-Doped Silica Particles for in Vivo Ratiometric Time-Gated Luminescence Imaging of Hypochlorous Acid	190
Synthesis and Luminescent Properties of $\text{La}_3\text{Si}_6\text{N}_{11}\text{:Ce}^{3+}$ Phosphors for White LEDs	191
The Enhancement of Structure Stability and Luminescence Intensity of $\text{LiYF}_4\text{:Ln}^{3+}$ Nanocrystals	192
Broad-Band Absorption and Sensitized Fluorescence of $\text{Sr}_8\text{MgY}(\text{PO}_4)_7\text{:Tb}^{3+}$ Phosphors by Codoping with Ce^{3+} Ions	193
Gradient Distribution of Dy in Diffusion Treated Nd-Fe-B Sintered Magnet	194

Optical Properties and Energy Transfer of Warm-White Light-Emitting $\text{Ca}_{12}\text{Al}_{14}\text{O}_{32}\text{F}_2$: Tb^{3+} , Eu^{2+} , Eu^{3+} Phosphors	195
Texture and Magnetism in Nanocrystalline $(\text{SmCo}_5)_{0.6}(\text{PrCo}_5)_{0.4}$ Magnets	196
The Comparative Study on Zeolite Y Modified by Rare Earth Metals	198
Fabrication and UV-Shielding Properties of Ceria/PA6 Film	199
$\text{Ni}_{1-x}\text{Mg}_x$ -SDC Anodes for Low-Temperature Solid Oxide Fuel Cells	200
Study on the Transformation of the Soil Erosion Types by Sm/Yb/Nd/Ce/La Tracing	201
Treatment of Ammonium-Nitrogen from Wastewater by Catalytic Oxidation with Ozone ..	202
Application of Rhamnolipids for Rare Earth La, Ce, Y, Eu Removal from Contaminated Soil by Washing	203
A Novel strategy for Synthesis of the CePO_4 Nano-Structure by Using the Ionic Liquid- Driven Supported Liquid Membrane System	204
Specificity for Extraction of Rare Earth Ions Using the Ionic Liquid-Based Tri- <i>n</i> - octylphosphine Oxide Systems	205
The Effect of Alkaline-Earth Metal Carbonates on Photoluminescence Property of β -Sialon: Eu^{2+}	206
Intense Triboluminescence and Crystal Structures of Eu(III) and Sm(III) Complexes with a Non-Discharge Mechanism	207
Structure and Luminescent Properties of Sm^{3+} -Doped $\text{Ca}_3\text{Bi}(\text{PO}_4)_3$ Phosphors	208
Multicolor Emitting Single Phase Phosphors for Plant Growth LEDs	209
Consequences of ET and MMCT on Luminescence of Ce^{3+} , Eu^{3+} and Tb^{3+} Doped LiYSiO_4	211
Multi-color Green-Blue Emission in $(\text{Ca}_{0.8}, \text{Ba}_{1.2})_{1-x}\text{Mg}_x\text{SiO}_4$: Eu^{2+} Phosphors	212
Study on the Decomposition of Bastnaesite by $\text{Ca}(\text{OH})_2$ -NaOH	213
Study on Recycle Technology for Waste Isotropic Bonded Nd-Fe-B Magnets	214
The Effects of Hydrogen Pressure on the Magnetocaloric Effect in $\text{LaFe}_{11.5}\text{Si}_{1.5}$ Compound	215
Investigations on the Roasting Process of Bayan Obo Tailings	216
Impurity Characterization by Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) in Terbium after Solid State Electrotransport Purification ..	217
Study on Improving the Recovery of Bayan Obo Oxidized Low-Intensity Magnetic Separation Tailings	218
Efficient Recycling of Lutecium Secondary Resources from LYSO Scraps Based on XAD Resin Containing Ionic Liquid System	219
Study on Recovery of Rare Earth and Iron from Tailing of Iron Ore Dressing in Baotou with Magnetizing Roasting	220
Separation of Rare Earth Metal Ions Using Novel Adsorbents Functionalized with Amic Acid	221

Study on Leaching of Rare Earth Oxide by H_2SO_4 and Defluorinating by Distillation	222
Quantitative Monitoring Ephemeral Gully Erosion Processes by Using Rare Earth Elements	223
Study on the Distribution Characteristics of Oil Particle Size of Oil-Containing Waste Water from Rare Earth Hydrometallurgy Process	224
Treatment of Magnesium Sulfate Wastewater of Rare Earth Transformation Using Oxalic Acid Precipitation Method	225
Graphene-Based Composites Used in the Separation of High-Purity Rare Earth and the Development of Intelligent Separation Device	226
Differential Expression of Proteins in Wheat Leaves Treated with Lanthanum	227
Ce^{3+} -Sensitized $\text{LiCa}_9\text{Gd}_{2/3}(\text{PO}_4)_7\cdot\text{Tb}^{3+}$ Phosphors: An Investigation on Luminescence and Energy Transfer	228
Effects of Preparation Conditions on Crystalline Phases of Aluminum and Tantalum Co-doped $\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$ Electrolytes	229
Influence of Cerium Reinforced Iron Ore Tailings Powder on the Growth of Bacteria	231
Luminescence Properties of Rare-Earth Ions ($\text{RE}^{3+} = \text{Sm}^{3+}$ and Eu^{3+}) in $\text{Y}_7\text{O}_6\text{F}_9$ Microrods Fabricated by Hydrothermal Method	232
Microstructure and Mechanical Properties of Graphene Reinforced $\text{TiC}/\text{Si}_3\text{N}_4$ Composite	233
$\text{NdBaCo}_{2-x}\text{Cu}_x\text{O}_{5+\delta}$ Cathodes for Low-Temperature SOFCs	234
Study on Rare Earth Electrolyte of Ba-Doped LSGM	235
Synthesis and Luminescent Properties of $\text{La}_3\text{Si}_6\text{N}_{11}:\text{Ce}^{3+}$ Phosphors for White LEDs	236
The Methane Catalytic Combustion of Ceria Composite Materials Modified with Tourmaline	237
Fusion Fuel Gas Storage Characteristics on La Activated ZrCo Intermetallic	238
Effect of Tb(III) on the Activity and Stability of Nattokinase	239
First-Principle Calculations and Re-analysis of the Optical Spectra and Electron Paramagnetic Resonance Parameters for Yb^{3+} in $\text{YAl}_3(\text{BO}_3)_4$ Crystal	241
Effect of Tb(III) on the Unfolding of Ciliate <i>Euplotes Octocarinatus</i> Centrin Induced by Guanidine Hydrochloride	242
INAA of U-Rich Samples for Lanthanides Using XRF-Based Internal Standard Method	244
Research on Alloying Technique of Mischmetal and Yttrium on Die-Casting Magnesium Alloy AZ91D	246
Geochemistry and Geologic Significance of Rare Earth Elements in Late Permian Coals from the Liupanshui Coalfield, Southwest China	247
Discussion on Environmental Impact Assessment of Rare Earth Mineral Industry	248
Effect of Neodymium on the Formation of the Craters Generated on the Al-17.5Si Alloy Surface Treated by High Current Pulsed Electron Beam	249

Effect of RE Element Nd and Thermal Treatment on Micro-structure and Mechanical Properties of A390 Al-Si Alloy	250
Effects of Aluminum Scrap (3004 Aluminum Alloy) on Structure and Properties of Zn-5Al-0.1RE Coating	251
Effects of RE on the Surface Morphology and Corrosion Resistance of Zn-23Al-0.3Si-2.0Mg Coating	252
Removal of La, Ce, Y and Eu from Soil by Saponin Biosurfactant	253
The Catalytic Combustion of Diesel Fuel over CeO ₂ / Tourmaline Composite Particles	254
The Purification Mechanism of Water Hyacinth on the Rare Earth Waste Water Pollutants ...	255
Research Progress of Rare Earth Perovskite-Type Catalyst in Methane Combustion	256
Luminescence Properties of Rare-Earth Ions (RE ³⁺ = Pr ³⁺ , Sm ³⁺ and Eu ³⁺) in Lu ₇ O ₆ F ₉ Microrods Fabricated by Hydrothermal Method	257

Section A: Full Paper

A High-Efficient System of the Ionic-Liquid Containing CHON-Type Extractant for Extraction and Recovery of Rare-earth Resources

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Abstract Our group focuses on the advanced field about developing novel “green” extractants (only C, H, O and N atoms), and the ionic liquid-mediated green extraction systems, which are applied in eco-friendly extraction and recovery of rare-earth resources. The diglycol amic acid derivative extractants were recently developed, which showed a high affinity for rare earth ions in ionic-liquid mediated extraction although a conventional and commercial phosphonic extractant did not. It is promising for effective recovery of the rare earth resources, such as Y, Eu, Tb, Nd, Pr, Dy and Lu, from waste phosphor powders, NdFeB magnets, and LYSO scraps. And it can be widely applied to supported liquid membrane system and silica gel/resin systems.

Keywords CHON-type extractant · Diglycol amic acid · Eco-friendly recycling · Ionic-liquid mediated extraction system · Rare-earth resources · Urban mine

1 Introduction

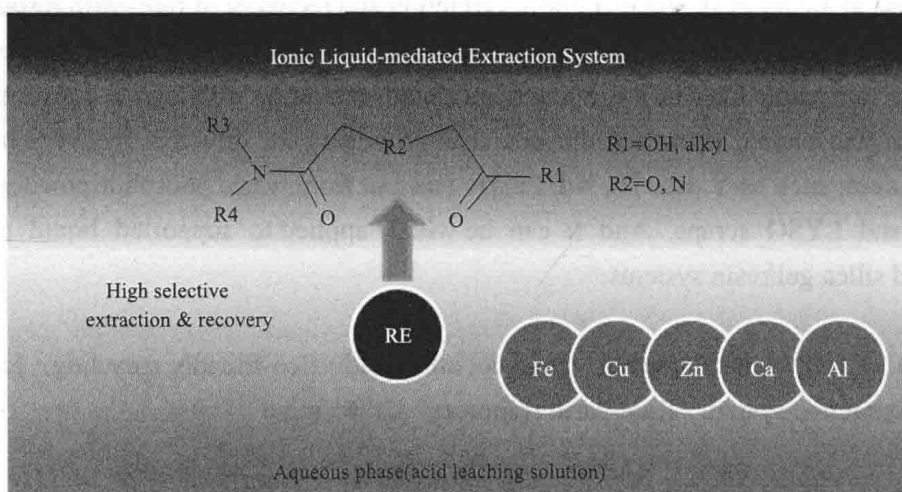
Currently, as new scarce strategic resources and from an economic growth point, the value of rare earths has been recognized with the rapid development of new industries such as the LED (light emitting diode) lighting industry, the electronic information industry, the automotive industry, etc. Furthermore, the price of rare earths has been increasing steadily. However, the output of rare earth resources has a strong regional dependence, in which China is dominant in the export of rare earth raw materials (about 90% market supply share), yet China rare earth output has been transforming from extensive availability to restricted availability since 2010, which is restricting the development of the above-mentioned new industries^[1].

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As an important nonrenewable natural resource, a stable supply of RE raw materials is crucial. In addition to mineral resource, secondary resource also has tremendous potential. An assessment of recycling potential global REs for the year 2020 is outlined, which includes magnetic storage media (Nd, Sm, Gd and Dy), lamp phosphors (Y, Ce, Eu and Tb) and so on^[2]. The recycling of “urban mines” has become an important source for sufficient supplies of RE materials. Thus, what is urgently needed in RE recycling is a highly selective and efficient extractant which can obtain individual REs and useful concomitant elements at the same time.

In recent years, ionic liquids have been highlighted as an eco-friendly alternative to organic solvents due to their unique properties such as negligible vapor pressure, non-flammability and high thermal stability. Their physicochemical properties are readily selectable, which make hydrophobic ionic liquids suitable for solvent extraction.

Recently, we reported efficient advanced extraction systems employing a novel extractant, DODGAA or DEHDGA, for the separation of rare earth metals from some transition metals such as Zn, Al and Fe in recycling processes^[3].



The diglycol amic acid derivative extractants DODGAA and DEHDGA, which are composed of only C, H, O, and N atoms and have lower solubility in the aqueous phase than conventional commercial extractants such as PC-88A and D2EHPA, were developed. Additionally, they showed some advantages not shared by the commercial extractants. For example, they were readily soluble in ionic liquids such as $[\text{C4mim}][\text{Tf}_2\text{N}]$ and the metal ions could be completely stripped from the IL extracting phase using a dilute acid such as H_2SO_4 , HCl or HNO_3 . In this article, we examined the extraction of a series of rare earth metal ions from the acid leaching solution using IL systems in the separation and recovery of the different types of rare earth secondary resources^[4].

2 Experimental

The recovery of rare earth metals from phosphor powders was examined. Leaching from a phosphor powder was carried out in two stages using the acid solution (5 M) as mentioned above. The first and second stage leaching solutions were prepared as feed solutions by diluting with sterile distilled water and adjusting the pH with 1 M NaOH solution. Extraction of metal ions from the feed aqueous solution to the IL [C4mim][Tf2N] phase containing desired concentration of DODGAA was performed. And performance evaluation of the diglycol amic acid DEHDGA functional XAD-type resin was carried out for the recycling and separation of the Lu (III) rare earth secondary resource from simulated leaching and real leaching solutions. These are discussed in terms of optimizing components of the recycling and separation process of Lu (III), such as the pH conditions of the mother liquor, solid-to-liquid ratio, extraction time, and operating temperature. Moreover, we also evaluated the recycle ability of the diglycol amic acid functional XAD-type resin. The molecular structures of extractants and the ILs used are shown in Fig. 1.

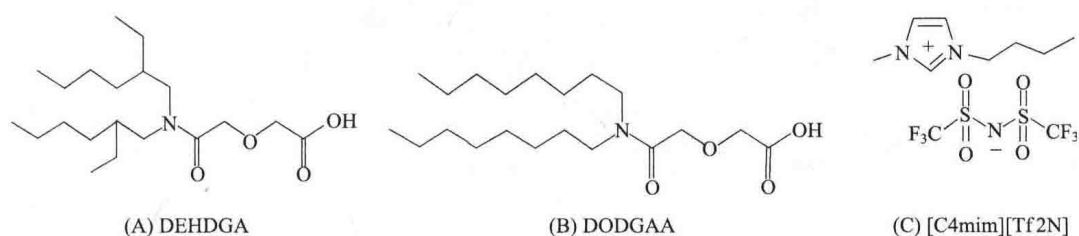


Fig. 1 Structures of diglycol amic acid derivative extractants [(A) and (B)] and the ionic liquid (C)

3 Results and Discussion

3.1 Recovery of rare earth metals from leaching solutions of waste phosphor powder

The practical application for recovery of rare earth metals are investigated using leaching solutions of waste phosphor powder by IL-based DODGAA system. As shown in Fig. 2, IL [C4mim][Tf2N] containing extractant DODGAA, which exhibited a high affinity for rare earth metal ions, was applied as an extracting solvent. The rare earth metals such as Y, Eu, La and Ce were extracted selectively against some metal impurities such as Fe, Al, Zn, whereas in the conventional PC-88a system, separation of rare earth metals from same metal impurities was difficult^[2]. Fig. 3 demonstrates the conceptual diagram based on the experimental results for the recovery of rare earth metals from waste phosphor powder of fluorescent lamps. The selective recovery of rare earth metals was possible from the leaching solutions by solvent extraction employing the IL-based DODGAA systems.

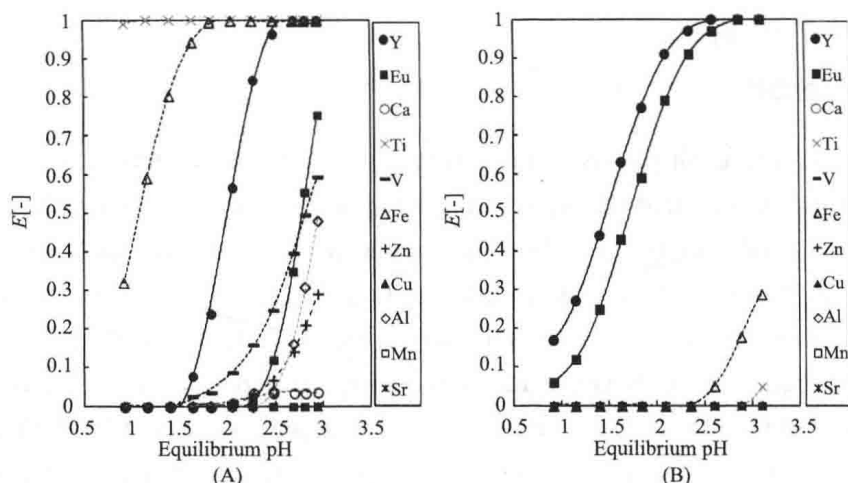


Fig. 2 Extraction behavior of metal ions dissolved in the simulated first-stage leaching solution: (A) PC-88A in *n*-dodecane system, (B) DODGAA in [C4mim][Tf2N] system. Feed phase: $[M_i] = 10$ ppm, $[SO_4^{2-}] = 0.05$ M; Extracting phase: [Extractant] = 10 mM, 30 °C

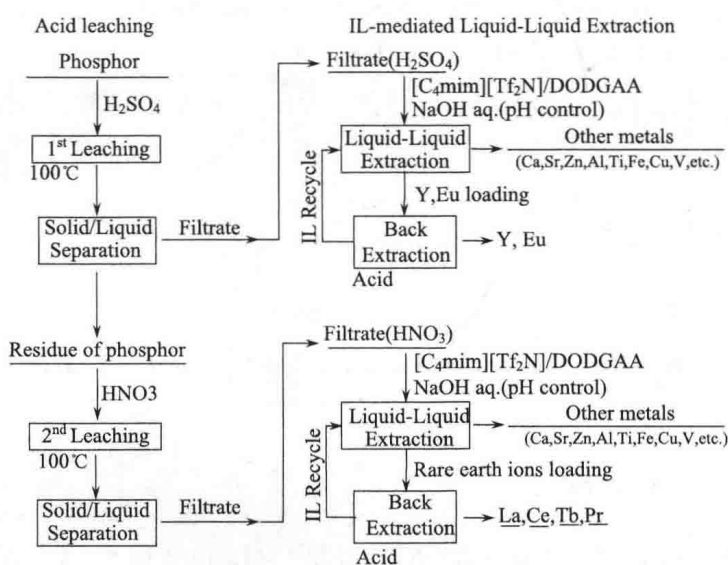


Fig. 3 Conceptual flowsheet for recovery of rare earth metals from waste phosphor powder of fluorescent lamps by wet process

3.2 Recovery of rare earth metals from leaching solutions of waste LYSO

Furthermore, recovery of high-value rare earth metals lutetium(Lu) are carried out from leaching solutions of waste LYSO using XAD-4 Resin-based DEHDGA-IL system. As shown in the following figures, XAD-4 Resin containing a diglycol amic acid derivative extractants DEHDGA and IL [C4mim][Tf2], which exhibited a high affinity for rare earth ions was used. Compared with that of the traditional CHON-type extractants, such as HTTA or CA-12, recovery rate of Lu(III) ions was significantly improved by using the DEHDGA-mediated resin system(Fig. 4). The conceptual flowchart for the process is shown in Fig. 5 which

includes microwave-assisted hot acid leaching and resin adsorption technology. The D₂EHDGAA functional XAD-type resin systems were prepared using the IL-immersion method, and [C₄mim][Tf₂N]-based D₂EHDGAA impregnated XAD-4 resin demonstrated not only a promising recycling performance for the Lu rare earth secondary resource, but also a satisfactory thermal separation stability performance.

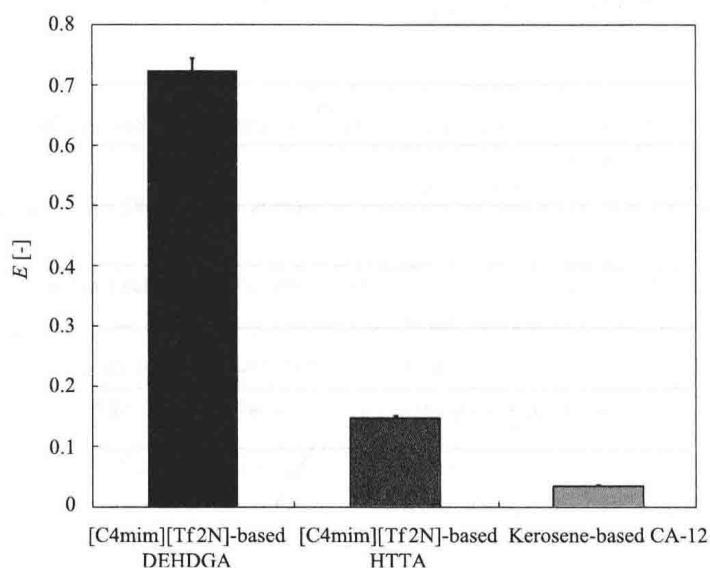


Fig. 4 The result of different CHON-type extractants for recycling of Lu rare earth secondary resources. The real leaching solution was used (Lu: 100 ppm and Y: 4.64 ppm): containing 50 mM of extractant, a solid-to-liquid ratio of 50 mg/ml, pH 1.4, and extraction time of 12 hrs at room temperature

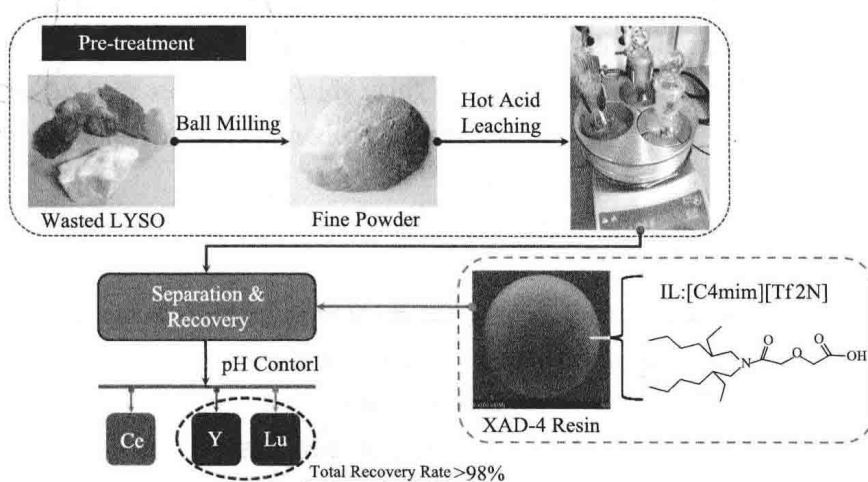


Fig. 5 Conceptual flowchart for recovery of Lu rare earth secondary resource from waste LYSO using a wet process and diglycol amic acid functional XAD-type resin

4 Conclusions

In future, the promising IL-based derivative extractant system will be applied to the practical recovery of rare earth secondary resources, especially for the recovery of Lu, in the waste and resources recovery industry.

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