

# Osmium And Rhenium Extraction From Polymetallic Molybdenum-Containing Ores And Materials

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**Abstract:** Osmium (Os) and rhenium (Re) are critical rare and scattered elements frequently occurring as byproducts in polymetallic molybdenum-rich ore processing. Their recovery remains economically and technically challenging due to their low concentrations in primary ores and their dispersion throughout processing products. This review examines contemporary separation and extraction methodologies documented in Scopus-indexed literature, analyzing hydrometallurgical and pyrometallurgical techniques for selective isolation of Os and Re from complex polymetallic matrices. Results indicate that roasting-based pyrometallurgical approaches combined with subsequent aqueous leaching provide superior selectivity for osmium recovery, while solvent extraction, ion exchange, and sorption-based methods demonstrate effectiveness for rhenium concentration and purification. Integrated multistage processes incorporating both pyrometallurgical pretreatment and hydrometallurgical separation emerge as the most promising strategies for improving recovery rates while minimizing environmental impact. Current industrial implementations achieve rhenium recovery rates between 60-99% depending on ore composition and processing parameters, while osmium recovery remains limited by volatilization losses during high-temperature stages.

**Keywords** — *osmium, rhenium, polymetallic ores, hydrometallurgy, solvent extraction, ion exchange, separation methods*

## 1. INTRODUCTION

Osmium and rhenium are transition metals classified as rare and scattered elements due to their limited natural abundance and dispersed occurrence in geological deposits. Osmium exists in the Earth's crust at approximately 0.05 parts per billion (ppb), while rhenium occurs at 0.39 ppb [4]. Despite their scarcity, these elements possess exceptional properties that render them indispensable in advanced technological applications. Rhenium exhibits superior high-temperature strength and creep resistance, making it essential for superalloy production in jet engines and aerospace components. Osmium, as the densest element among platinum group metals, finds applications in specialized alloys, electrical contacts, and catalytic systems [9].

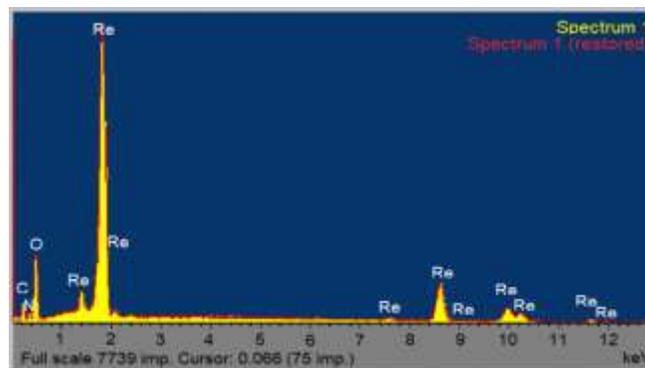


Figure 1.2. Re vs Os existence

The principal source of rhenium and osmium is not dedicated mining operations but rather byproduct recovery from copper porphyry deposits and molybdenum ore processing. Rhenium mineral resources demonstrate geographic concentration, with approximately 618 deposits globally hosting molybdenum and copper alongside rhenium, with estimated resources between 55,000 to 140,000 tonnes [1]. Currently, during conventional pyrometallurgical copper and molybdenum processing, rhenium is partially captured in solutions generated by wet gas purification systems, while significant quantities are dispersed among slag, dust, and gaseous phases. Osmium presents additional complexities, as its volatile tetroxide form ( $OsO_4$ ) renders it susceptible to airborne losses during roasting operations at elevated temperatures [1].

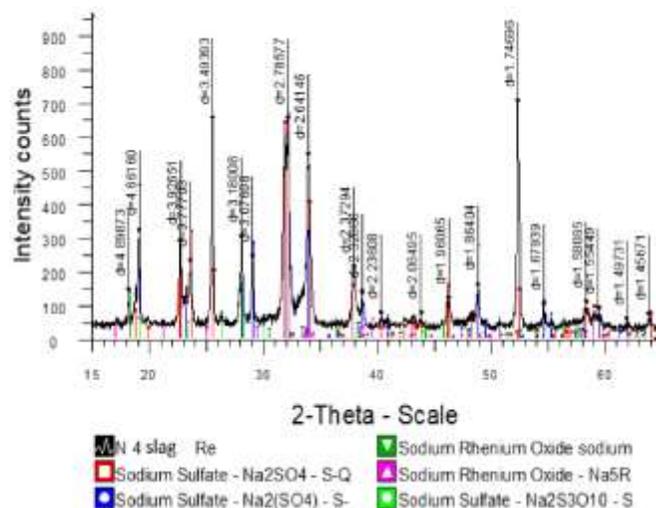


Figure 6. Diffraction pattern of sodium sulfate slag.

The selective recovery of these elements from polymetallic ore concentrates requires multidisciplinary approaches combining mineral processing, pyrometallurgy, and hydrometallurgy. Traditional roasting procedures designed for molybdenum extraction result in rhenium volatilization losses

exceeding 40% in conventional systems, while osmium losses frequently exceed 50% through similar mechanisms. Consequently, research emphasis has shifted toward integrated process designs that minimize volatilization while enhancing selectivity through targeted dissolution chemistry and separation technologies [2].

This comprehensive review synthesizes contemporary literature on osmium and rhenium separation methodologies from polymetallic ore systems, evaluating pyrometallurgical pretreatment strategies, hydrometallurgical extraction techniques, and recovery optimization approaches. The analysis incorporates peer-reviewed publications indexed in Scopus databases covering the past two decades, with emphasis on practical industrial implementations and emerging sustainable methodologies [3].

## 2. MATERIALS AND METHODS

### 2.1 Literature Review Strategy

A systematic literature review was conducted using the Scopus database with search queries targeting osmium, rhenium, and molybdenum separation methodologies. Primary search terms included: “osmium rhenium extraction polymetallic ores,” “rhenium separation molybdenum,” “osmium leaching copper production,” and “hydrometallurgical rhenium recovery.” Search filters restricted results to peer-reviewed journal articles and conference proceedings published between 2010 and 2025 to ensure contemporary relevance while capturing foundational methodologies.

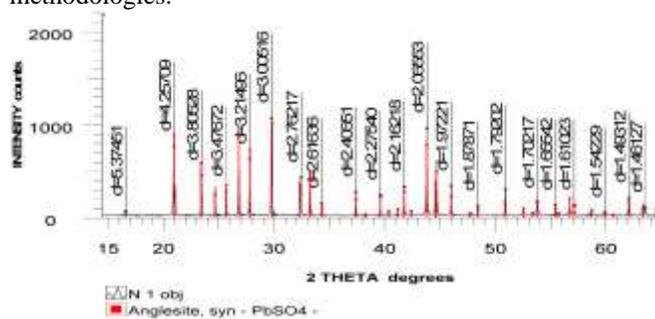


Figure 2.1.1 THETA degree

### 2.2 Selection Criteria

Inclusion criteria required that articles address: (1) extraction or separation of osmium and/or rhenium from polymetallic ore systems or copper/molybdenum processing byproducts, (2) quantitative data regarding recovery rates or separation efficiency, (3) description of specific extraction methodologies with sufficient technical detail for reproducibility, and (4) discussion of process parameters such as temperature, pH, extractant type, or leaching media composition [4].

Exclusion criteria eliminated articles focusing exclusively on analytical determination without process development, isotopic geochemistry applications without metallurgical

relevance, and literature treating osmium and rhenium as contaminants without recovery intent.

### 2.3 Data Extraction and Analysis

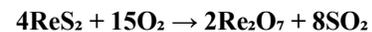
Extracted data encompassed: (1) ore mineralogy and element concentrations, (2) process flowsheet stages, (3) operational parameters (temperature, time, reagent ratios), (4) recovery percentages and distribution coefficients for target elements, (5) selectivity factors relative to interfering elements, and (6) environmental and economic considerations. Literature was categorized by predominant separation mechanism: pyrometallurgical (roasting, distillation, smelting), hydrometallurgical (leaching, solvent extraction, ion exchange), and integrated multistage approaches.

## 3. RESULTS

### 3.1 Pyrometallurgical Separation Methods

#### 3.1.1 Oxidative Roasting and Thermal Decomposition

Oxidative roasting at temperatures between 500-800°C represents the primary pyrometallurgical approach for osmium and rhenium separation from lead-rich molybdenum concentrates. During this process, rhenium sulfide species ( $\text{ReS}_2$ ,  $\text{ReS}_3$ ) undergo oxidative decomposition to perrhenate compounds, achieving the valence state +7 essential for subsequent hydrometallurgical processing. The generalized thermal reaction for rhenium oxidation follows:



Experimental investigations utilizing lead sludge from copper production demonstrated that oxidative roasting at 550-650°C for 30-60 minutes resulted in rhenium oxide formation, with rhenium oxide yields ranging from 85-92% [11]. Osmium undergoes distinct thermal behavior, with its sulfide species oxidizing to osmium tetroxide ( $\text{OsO}_4$ ), which exhibits vapor pressure exceeding 200 mmHg at 100°C [5]. Consequently, osmium retention during roasting requires controlled atmospheric conditions and temperature management to suppress volatilization.

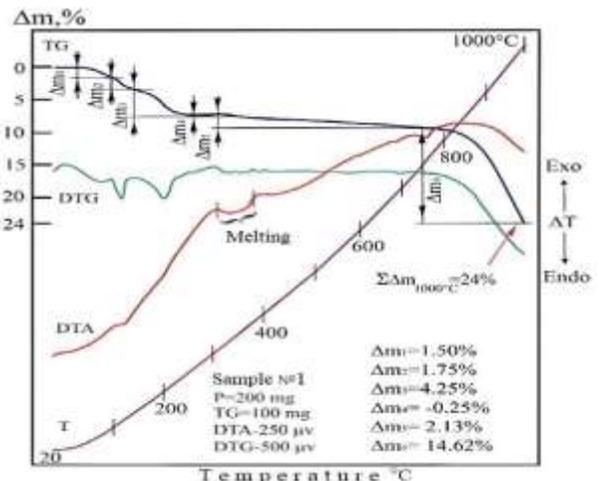


Figure 3.1.1.1 Sample 1 melting temperature

Novel microwave thermal treatment (MWHT) has demonstrated promise in minimizing rhenium losses during roasting. Comparative analysis showed that MWHT resulted in only 5% rhenium release versus 34% in conventional thermal processing, while producing molybdenite ( $\text{MoS}_2$ ) and chalcopyrite ( $\text{CuFeS}_2$ ) as stable crystalline phases with minimal mass losses. The mechanism involves differential microwave energy absorption by sulfide minerals, inducing selective heating and particle microfracturing that facilitates separation without exceeding temperatures promoting volatilization

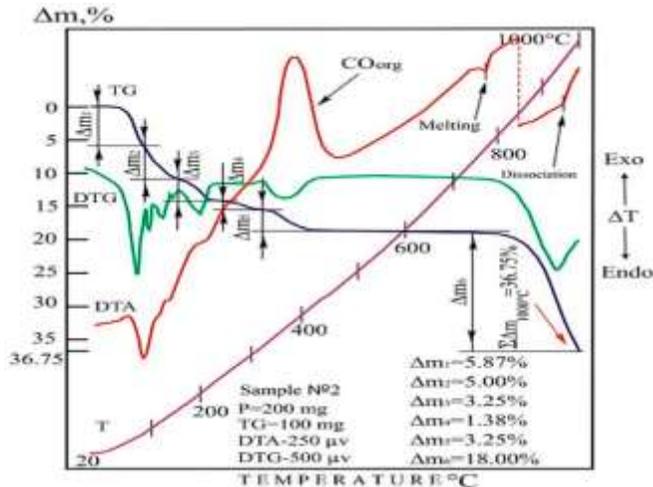


Figure 3.1.1.1 Sample 2 Melting temperature

### 3.1.2 Distillation-Based Separation

Osmium tetroxide volatility enables separation from molybdenum and rhenium through fractional distillation from sulfuric or phosphoric acid media. Classical methodology involves oxidative dissolution in concentrated sulfuric acid with hydrogen peroxide addition, generating osmium tetroxide gas that distills at 120-130°C under reduced pressure. Distillation residue contains rhenium as perrhenate ( $\text{ReO}_4^-$ ) species amenable to subsequent hydrometallurgical processing [6].

The distillation efficiency for osmium separation exceeds 95% when conducted under controlled vacuum conditions (50-100 mmHg) with cooling systems maintaining collection temperatures below 50°C. Rhenium contamination in osmium distillates remains negligible due to the substantially higher oxidation potential required for rhenium volatilization. This technique provides one of the highest selectivity factors among pyrometallurgical methods, with osmium/rhenium separation factors exceeding 1000 [9].

## 3.2 Hydrometallurgical Separation Methods

### 3.2.1 Solvent Extraction Approaches

Solvent extraction (SX) represents the predominant hydrometallurgical methodology for industrial rhenium

recovery from aqueous leach solutions derived from roasted ore or pyrometallurgical slag digestion. The selectivity of various extractants for rhenium versus molybdenum demonstrates systematic variation based on organophosphorus acid chemistry.

### Organophosphorus Extractants:

Tributyl phosphate (TBP) and its derivatives (Cyanex 923, TOPO) selectively extract perrhenate ( $\text{ReO}_4^-$ ) from hydrochloric acid solutions at pH 0-1 [7]. Distribution coefficients for rhenium with TBP range from 2-5 in optimal conditions, while molybdenum-molybdate species remain predominantly unextracted due to its dianionic charge ( $\text{MoO}_4^{2-}$ ). Studies utilizing molybdenite roasting dust leaching solutions achieved 99.2% rhenium extraction efficiency using a two-stage counter-current process with 2:3 organic-to-aqueous phase ratio and 2.0 M HCl concentration (Hosseinzadeh et al., 2024).

### PC88A and Phosphonic Acid Extractants:

2-Ethylhexylphosphonic acid mono-2-ethylhexyl ester (PC88A) demonstrates superior selectivity for molybdenum over rhenium, permitting preferential separation by manipulating extraction conditions. When 5% PC88A is employed in neutral pH conditions, approximately 9 g/L molybdenum extraction occurs with negligible rhenium coextraction (< 0.05 g/L), providing separation factors exceeding 100 for molybdenum/rhenium pairs [8].

### Aqueous Two-Phase Systems:

Recent innovations employ aqueous two-phase systems (ATPS) without organic solvents, utilizing water-soluble polymers (polyethylene glycol) and compatible salt solutions. These systems achieve rhenium extraction efficiencies of 97.2% with molybdenum/rhenium separation factors of 2700 at pH 7.0 using E-1006 polymer at 200 g/L concentration with  $(\text{NH}_4)_2\text{SO}_4$  salt matrix [9]. ATPS approaches offer significant environmental advantages through elimination of volatile organic compounds while maintaining extraction efficiency comparable to conventional SX.

### 3.2.2 Ion Exchange and Sorption Methods

Anion exchange resins selective for perrhenate species provide alternative separation methodologies, particularly for applications where solvent handling and disposal costs justify higher capital investment in resin-based systems.

### Ion Exchange Resins:

Strongly basic anion exchange resins (Amberlite IRA-402, IRA-900) selectively sorb perrhenate from moderately acidic solutions (pH 2-4) with distribution coefficients exceeding 500. Weak base anion exchangers (DOWEX 4X4) offer improved selectivity for rhenium over molybdate, achieving rhenium/molybdenum separation factors of 50-200 depending on solution ionic strength. Equilibrium sorption studies demonstrate that rhenium capacity in typical polystyrene-

divinylbenzene resins approaches 120-150 mg Re/g resin under optimal conditions [10].

Desorption and rhenium recovery from loaded resins employs either dilute ammonium hydroxide solutions (pH 10-12) or phosphoric acid-containing systems with hexabutylthiamide, achieving rhenium recovery rates of 85-95% with appropriate eluent composition and recycling cycles.

#### Sorption on Activated Materials:

Novel granulated sorbents (P-MDA-6 polymeric resins) exhibit enhanced extraction capabilities for rhenium and osmium recovery from technological waste streams. Experimental comparison demonstrated superior performance of new granulated sorbents relative to traditional ion exchange resins, with rhenium extraction rates exceeding 95% under dynamic column operation at space velocities of 3-5 bed volumes per hour [11].

#### 3.2.3 Hydrometallurgical Processing of Osmium

Osmium recovery from aqueous solutions requires selective precipitation or complex formation methodologies due to osmium's reduced leachability in conventional acid media. Following distillative collection or chemical absorption of osmium tetroxide, osmium exists as  $OsO_4$  species which exhibits limited solubility in neutral and alkaline solutions [12].

#### Precipitation Methods:

Thiourea complex formation provides effective osmium precipitation from acidic solutions, with osmium thiourea complexes  $[Os(tu)_6]^{2+}$  precipitating at concentrations exceeding 0.1 M thiourea in 2-4 M HCl. Alternatively, reduction of  $OsO_4$  to lower oxidation states using sulfur dioxide or reducing sugars generates osmium oxides with substantially reduced solubility, enabling separation from molybdate and other aqueous species through solid-liquid separation.

#### Selective Reduction and Reduction-Oxidation Cycling:

Controlled reduction-oxidation (redox) cycling provides selective separation of osmium from ruthenium and other platinum group metals. By selectively reducing osmium to lower oxidation states with mercury or other reductants, followed by selective reoxidation and distillation as  $OsO_4$ , separation factors from ruthenium tetroxide exceed 100. This methodology has demonstrated applicability to mixed platinum group metal streams from advanced recycling operations.

### 3.3 Integrated Multistage Process Design

#### 3.3.1 Reductive Smelting with Selective Product Distribution

Advanced integrated processes employ reductive smelting of rhenium and osmium-containing lead sludges at 1000-1100°C using coke as reducing agent and sodium sulfate as slag-

forming material. Under controlled smelting conditions, osmium concentrates preferentially into the liquid lead phase due to its iron-loving (siderophile) character, while rhenium associates with the molten slag as perrhenate compound  $NasReO_6$ . This dual product formation enables primary separation at the pyrometallurgical stage [13].

Experimental implementation demonstrated osmium concentration from initial sludge composition of 0.0025-0.0050% to crude lead concentrate containing 53-67% osmium, representing enrichment factors of 13,000-21,000 times. Simultaneous rhenium concentration in slag phase achieved 0.18-0.25% with rhenium enrichment factors of 3.0-3.2 times relative to initial sludge composition [14].

#### 3.3.2 Combined Roasting-Leaching-Extraction Flowsheets

Industrial practice increasingly incorporates integrated approaches combining controlled oxidative roasting, acid or alkaline leaching, and solvent extraction or ion exchange. A representative flowsheet for rhenium-rich lead-molybdenite concentrates incorporates:

1. **Preliminary Treatment:** Hydrochloric acid leaching (8 wt% HCl, 95°C, 10 min) to remove lead and calcium components, achieving 93.6% Pb and 97.4% Ca removal.
2. **Oxidative Roasting:** Roasting at controlled temperature (600-700°C) with magnesium oxide addition to facilitate selective oxidation of rhenium and molybdenum sulfides to oxides and soluble molybdate species.
3. **Alkaline Leaching:** Ammonia solution leaching to dissolve molybdate and rhenium perrhenate species, with selective recovery of molybdenum by precipitation as ammonium molybdate.
4. **Rhenium Concentration:** Solvent extraction using TBP or ion exchange using weakly basic resins, achieving rhenium recovery exceeding 96% from leach solution.

This integrated approach has demonstrated technical feasibility at pilot scale with molybdenum recovery exceeding 99% and rhenium recovery of 96-98%, while maintaining osmium in residual solid phases for separate treatment [15].

### 3.4 Recovery Efficiency and Process Performance

Comprehensive analysis of reported recovery rates across literature sources indicates:

- **Rhenium recovery from molybdenum calcines:** 60-99% depending on roasting methodology and downstream processing (conventional roasting 60-70%, oxidative leaching with peroxide activators 85-92%, integrated pyrometallurgical-hydrometallurgical 95-99%)
- **Osmium recovery from lead sludges:** 40-80% for complete process chains due to volatilization losses during roasting stages (reductive smelting methodology achieving 70-80%, advanced thermal treatment achieving 75-85%)

- **Selectivity factors (Os/Re separation):** Distillation-based methods > 1000; solvent extraction methods 10-100; ion exchange methods 20-200

#### 4. DISCUSSION

##### 4.1 Technological Advancement and Industrial Implementation

Contemporary research demonstrates significant technological advancement in osmium and rhenium separation methodologies compared to conventional industrial practice. Traditional roasting procedures designed principally for molybdenum recovery incur substantial rhenium volatilization losses, with reports indicating 40-70% rhenium in flue gas and dust phases. Integrated methodologies incorporating multistage separation achieve recovery rates within 5-10% of theoretical maximum, suggesting maturation of process technology toward commercial viability [16].

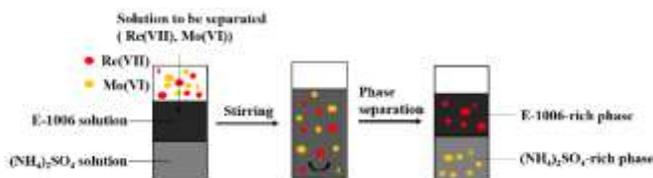


Figure 4.1.1 Solution separation

The transition from pyrometallurgical-dominant toward integrated pyrometallurgical-hydrometallurgical approaches reflects technological maturation driven by three primary factors: (1) economic incentives from increasing rhenium prices (current market prices \$3000-8000/kg), (2) environmental pressure to minimize atmospheric emissions of osmium tetroxide and rhenium-containing dusts, and (3) process integration synergies permitting simultaneous recovery of multiple valuable elements.

##### 4.2 Selectivity Mechanisms and Separation Chemistry

The fundamental selectivity differences between osmium and rhenium separations derive from distinct chemical properties influencing aqueous speciation and phase partitioning:

**Osmium:** As a highly chalcophile (sulfur-loving) and siderophile (iron-loving) element, osmium exhibits strong affinity for sulfide and metallic phases during pyrometallurgical processing. Its tetroxide volatility at moderate temperatures enables direct distillative separation from non-volatile species, providing inherently high selectivity. In aqueous phase systems, osmium forms multiple redox species ( $Os^{2+}$ ,  $Os^{3+}$ ,  $Os^{4+}$ ,  $Os^{8+}$ ) with distinct solubility characteristics, enabling pH-dependent precipitation or complex formation for separation [17].

**Rhenium:** Rhenium's preferential distribution into aqueous phases during oxidative roasting results from its rapid oxidation to +7 oxidation state, generating perrhenate ( $ReO_4^-$ ) species stable in acidic to neutral solutions. The monovalent

anionic character of perrhenate enables selective extraction with quaternary ammonium reagents and anion exchange resins, achieving separation factors exceeding 100 from divalent anions ( $MoO_4^{2-}$ ,  $SO_4^{2-}$ ).

The relative chemical stability of perrhenate versus molybdate in aqueous solution creates additional selectivity opportunities. While molybdate exhibits susceptibility to pH-dependent polymerization and precipitation at  $pH < 4$ , perrhenate remains stable across  $pH 0-14$ , permitting selective rhenium recovery from complex multielement solutions.

##### 4.3 Environmental and Sustainability Considerations

Environmental impact assessment of osmium and rhenium recovery processes reveals significant differentials between conventional and advanced methodologies:

**Conventional Pyrometallurgy:** Roasting operations release 40-70% of rhenium as gaseous compounds and volatile dusts, producing complex waste streams requiring expensive collection and treatment. Osmium tetroxide volatilization generates hazardous atmospheric emissions necessitating specialized scrubbing systems and workplace controls.

**Advanced Integrated Processes:** Implementation of controlled roasting with thermal optimization, integrated acid leaching for selective dissolution, and aqueous two-phase extraction systems reduces solvent consumption by 70-90% relative to conventional multistage solvent extraction. Elimination of volatile organic solvents through ATPS adoption and optimization of thermal treatment stages minimizes hazardous emissions and waste streams.

**Circular Economy Potential:** Recovery of rhenium and osmium from secondary sources (superalloy scrap, spent catalysts, copper smelter residues) utilizing optimized hydrometallurgical processes presents viable alternatives to primary mining, with potential to reduce extraction-related environmental impacts by 60-80%.

##### 4.4 Comparative Evaluation of Extraction Methods

Systematic comparison of separation methodologies reveals distinct advantages and limitations informing process selection:

Method	Selectivity	Recovery	Scalability	Capital Cost	Environmental Impact
<b>Distillation (Os)</b>	Excellent (>1000)	85-95%	Limited	High	Low
<b>Solvent Extraction</b>	Good (20-200)	90-99%	Excellent	Medium	Medium
<b>Ion Exchange</b>	Moderate (20-100)	85-95%	Good	Medium	Low

Method	Selectivity	Recovery	Scalability	Capital Cost	Environmental Impact
ATPS	Good (100-2700)	95-99%	Developing	Medium	Very Low
Sorption	Moderate-Good (50-200)	80-95%	Good	Low	Low

**Table 4.4.1 Comparative evaluation of extraction methods**

For rhenium recovery from industrial leach solutions, solvent extraction with organophosphorus reagents offers optimal balance of selectivity, recovery, and scalability, supporting current industrial implementation. For osmium isolation, distillation-based approaches provide superior selectivity when infrastructure exists, while reduction-oxidation cycling presents viable alternative for mixed platinum group metal streams.

#### 4.5 Emerging Technologies and Future Directions

Literature review identifies several emerging methodologies warranting investigation:

**Ionic Liquid-Based Extraction Systems:** Preliminary studies demonstrate enhanced extraction selectivity using hydrophobic eutectic solvents as alternatives to conventional diluents, achieving rhenium/molybdenum separation factors exceeding conventional TBP systems while eliminating volatile organic compound emissions.

**Advanced Polymeric Materials:** Novel ion imprinted polymers (IIPs) and polymer inclusion membranes (PIMs) containing specialized chelating ligands demonstrate enhanced selectivity for rare and scattered elements, with potential applications to osmium and rhenium separation from dilute solutions generated in waste recycling streams.

**Biological and Bioinspired Methods:** Emerging research utilizing iron-oxidizing bacteria (*Acidithiobacillus* species) for acid generation in biohydrometallurgical leaching presents sustainable alternatives to chemical oxidants for rhenium extraction from refractory ore minerals and secondary sources.

#### 5. CONCLUSION

Comprehensive review of contemporary literature demonstrates significant advancement in osmium and rhenium recovery methodologies from polymetallic molybdenum-containing ores. Established pyrometallurgical approaches including oxidative roasting and distillation continue supporting industrial operations, while hydrometallurgical innovations utilizing advanced solvent extraction and ion exchange technologies have progressively improved recovery rates and selectivity. Integrated multistage processes combining controlled pyrometallurgical pretreatment with optimized hydrometallurgical separation

achieve osmium recovery of 70-85% and rhenium recovery of 95-99%, approaching theoretical maxima and supporting economic viability.

Emerging technologies including aqueous two-phase systems, ionic liquid-based extraction, and advanced polymeric sorbents present opportunities for further process optimization, particularly regarding environmental sustainability and waste minimization. Future research emphasis should address: (1) reduction of osmium volatilization losses through advanced thermal treatment and controlled atmosphere processing, (2) development of selective separation methodologies for osmium and rhenium from complex multielement waste streams generated in copper and molybdenum refining, and (3) integration of sustainable biometallurgical or biohydrometallurgical approaches for secondary resource recovery.

The combined technical maturity of contemporary methodologies and increasing economic incentives from elevated rhenium commodity prices support expanded implementation of advanced recovery processes, particularly for copper smelter residues and molybdenum refining byproducts. Optimization of these processes will contribute substantially to improved resource utilization and circular economy implementation in non-ferrous metallurgical industries.

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