

Review

Recovery of Valuable Metals from Spent HDS Catalysts and Comprehensive Utilization of Spent HDS

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Abstract

Hydrodesulfurization (HDS) is a critical technology for producing clean petroleum products. With the steady growth in annual crude oil processing volumes, the consumption of HDS catalysts has been continuously increasing. The massive release of spent catalysts imposes significant pressure on both the environmental compliance and sustainable operation of companies. Spent HDS catalysts contain relatively high concentrations of valuable metals such as Mo, Ni, V, and Co. These materials are toxic and harmful heavy metal pollutants, yet they also constitute an important secondary resource of strategic metals with extremely high recycling value. Based on a systematic comparison of relevant recovery technologies, this paper reviews the current status of valuable metal recovery from spent HDS catalysts. It summarizes typical process routes for the full-component recovery of valuable metals and discusses future research directions, with the aim of providing a theoretical reference for the high-value resource utilization of spent HDS catalysts.

Keywords: spent HDS catalysts; resource recovery; hydrometallurgical leaching

1. Introduction

Petroleum serves as the fundamental energy source of the modern industrial system, underpinning the operation of key industries worldwide. Hydrodesulfurization (HDS), hydrodenitrogenation, and hydrocracking are the core technologies for crude oil refining and clean fuel production [1,2]. Catalysts play a vital role in removing sulfur, carbon, and oxygen-containing compounds, as well as unsaturated hydrocarbons, from petroleum, thereby reducing environmental pollution and improving fuel quality [3]. Over 90% of the reactions in petroleum processing rely on catalysts [4,5]. The total crude oil consumption in China over the past decade is shown in Figure 1. As crude oil consumption rises, the volume of spent catalysts generated increases accordingly. According to petrochemical industry data, approximately 0.354 kg of spent catalyst is produced per ton of crude oil refined [6]. In 2025, China's crude oil processing volume was approximately 762 million tons, resulting in roughly 0.27 million tons of spent catalyst. The cumulative output of spent petroleum catalysts in China over the past ten years has reached 2.42 million tons, while the global petroleum industry discards as much as 1.7×10^5 tons of spent catalysts annually [7].



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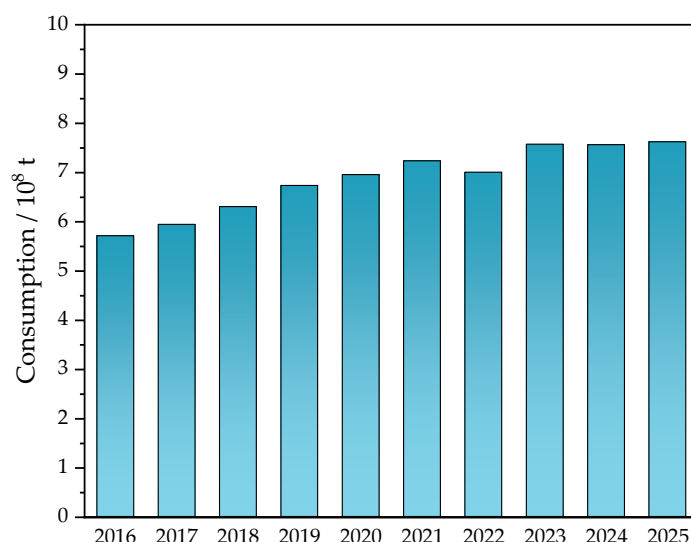


Figure 1. China's total crude oil consumption in the decade 2016–2025.

The surface area of fresh HDS catalysts is about 219–234 m²/g [8] and they are pale green [9]. After prolonged use, the catalysts gradually deactivate and are discarded, typically appearing black or dark gray. Compared with fresh catalysts, spent HDS catalysts have a lower surface area of about 100–200 m²/g and a reduced pore volume of approximately 0.66–1.1 mL/g [10]. HDS catalysts are cylindrical extrudates with columnar pore channels, and the particle size mostly ranges from 5 to 6 mm [11]. Upon activation, sulfur active sites narrow the pore channels, and during extended operation, continuous coke deposition further blocks the pores, reducing the gas adsorption capacity. Consequently, spent catalysts experience significant pore volume loss and a corresponding decrease in specific surface area [12], with the particle size diminishing to approximately 1 mm [13,14]. This densification greatly increases the diffusion resistance of reactants into the particle interior, posing considerable challenges for the subsequent recovery of valuable elements.

HDS catalysts typically use Al₂O₃ as a support and contain various heavy metals and acidic oxides. Direct discharge or large-scale stockpiling can easily lead to the leakage of hazardous substances, endangering the ecological environment and human health; as such, it has been classified as hazardous solid waste [15,16]. The reuse path of spent HDS catalysts depends on the extent of deterioration. When metal contaminant deposition is relatively low, catalyst activity can be restored through carbon removal, desulfurization, and impurity elimination, allowing reuse in petroleum HDS. However, during high-temperature regeneration, active metals (Ni or Co) react readily with the Al₂O₃ support to form spinel phases. Therefore, the activity of regenerated catalysts is generally lower than that of fresh catalysts [10]. Marafi et al. [14] continuously added ferric nitrate solution to an oxalic acid leaching system for spent HDS catalysts and found that the approach improved V removal and enhanced specific surface area, pore volume, and HDS activity. After carbon removal, catalyst activity was restored to over 95%. Ahn et al. proposed a remanufacturing process for deactivated spent HDS catalysts including oil washing, complete incineration, and acid leaching [17]. This process successfully removed coke, sulfur, and vanadium contaminants. It recovered 75% of the original HDS activity and 85% of the mechanical strength. However, the oxalic acid leaching step caused a substantial loss of active metals. During prolonged service, vanadium continuously accumulates and forms VS_x and mixed nickel–vanadium sulfides. A significant fraction of the active metals agglomerate and sinter, converting into sulfides such as MoS₂ and NiS, which block the support pore channels. Regeneration under these conditions is difficult, and extracting the valuable metals becomes the preferred route for high-value resource recovery [18]. Some researchers have

proposed using spent catalysts directly, after detoxification, as raw materials for cement manufacturing or construction materials [10]. For example, Romero [19] detoxified spent HDS catalysts and used the treated material as a substitute for natural mineral feedstocks in ceramic tile production, thereby achieving low-cost reuse. Gao et al. [20] employed the ash obtained from calcining spent HDS catalysts as an additive catalyst in the pretreatment deoiling stage. The active metal oxides in the ash improved the pyrolysis performance of the oil-laden catalyst, enabling the closed-loop utilization of spent HDS catalysts. However, direct disposal and reuse are only suitable when the hazardous constituents in the spent catalyst are present at relatively low levels.

Spent HDS catalysts contain metals such as Mo, Ni, V, and Co, which are extremely scarce in the Earth's crust and are referred to as "Energy Critical Metals" [21,22]. Their concentrations in spent catalysts are significantly higher than those in primary mineral resources [23]. Therefore, recovering these valuable metals is of great significance in alleviating the shortage of energy metals. Unlike existing reviews, which typically focus on a single recovery method or a single target metal, this review is structured around the full metal recovery process: roasting–hydrometallurgical leaching–solvent extraction. The extraction and separation techniques specific to Mo, Ni, V, Co, and Al are systematically analyzed. Incorporating economic feasibility and environmental impact considerations, the review summarizes integrated process routes for the full-component recovery of valuable metals, aiming to provide a theoretical reference for the high-value utilization of spent HDS catalysts.

2. Main Phase Composition of Spent HDS Catalysts

The chemical composition of spent HDS catalysts varies significantly based on the source, operating conditions, and service life. Typical compositions are shown in Table 1. HDS catalysts commonly used in industry are predominantly Co-Mo/ γ -Al₂O₃ or Ni-Mo/ γ -Al₂O₃ [22], with γ -Al₂O₃ typically serving as the support and accounting for the high Al content. Active components include valuable elements such as Mo, Ni, V, and Co, while elements like N, C, and P are incorporated to improve catalytic performance. After extended use, spent catalysts generally accumulate substantial amounts of carbon and sulfur and may contain minor phosphorus impurities. In fresh HDS catalysts, the active metals are present as NiO, MoO₃, and CoO [18,24], as shown in Table 2. The Mo, Ni, V, and Co in spent HDS catalysts, however, are primarily present as sulfides, composite sulfides, and oxides.

Table 1. Main chemical compositions of spent hydrodesulfurization (HDS) catalysts. (wt.%).

	Types	Mo	Al	Ni	V	Co	S	C	P	Reference
1	Spent HDS catalysts	11.40	28.20	2.55	-	-	7.18	6.81	2.21	[25]
2	Ni-Mo/ γ -Al ₂ O ₃	4.33	23.30	13.24	5.89	-	5.11	-	-	[23]
3	Ni-Mo/ γ -Al ₂ O ₃	11.50	23.10	2.10	-	-	8.30	2.80	-	[26]
4	Mo-Ni/Al ₂ O ₃	11.60	27.10	2.50	-	-	9.70	4.80	1.90	[27]
5	Mo-Ni/Al ₂ O ₃	17.37	25.41	2.80	-	0.06	8.11	-	1.88	[28]
6	Mo-V/Al ₂ O ₃	4.69	17.21	4.83	13.62	-	9.51	-	0.37	[29]
7	Ni-Mo/Al ₂ O ₃	12.10	30.00	2.60	<0.04	<0.01	-	-	-	[30]
8	Co-Mo/Al ₂ O ₃	12.00	26.00	0.91	<0.04	1.70	-	-	-	[30]
9	Co-Mo/Al ₂ O ₃	8.70	19.20	0.10	0.25	2.60	-	-	1.50	[15]
10	Mo-Co-Ni/Al ₂ O ₃	9.35	37.42	1.72	-	2.18	0.73	13.71	0.28	[31]

Table 2. Phase composition of metal elements.

Elements			Phases		
Mo	MoS ₂	Mo ₇ S ₈	MoS ₂ , MoO ₃	MoS ₂ , MoO ₂ ,	Mo ₃ O
Ni	NiV ₂ S ₄	Ni ₃ S ₄ , NiV ₃ S ₆	NiS	-	NiO
V	V ₂ S ₃ , V ₂ O ₅	V ₃ S ₄	V ₃ S ₄	V ₂ S ₃ , VO ₂	-
Co	-	-	-	-	CoO
Al	γ-Al ₂ O ₃	Al ₂ O ₃	γ-Al ₂ O ₃	γ-Al ₂ O ₃	Al ₂ O ₃
Reference	[32]	[29]	[33]	[34]	[24]

3. Recovery of Valuable Metals from Spent HDS Catalysts

Based on the chemical properties of spent HDS catalysts, chemical methods are used to separate valuable metal elements from the spent material. A typical overall process consists of three parts: pretreatment of spent catalysts, leaching of valuable metals, and selective separation.

3.1. Pretreatment

Spent HDS catalysts are usually covered in large amounts of residual oil, carbon deposits, sulfur and other contaminants. During direct leaching, these substances hinder contact between the leaching reagent and the target metal, reducing metal extraction efficiency. The residual oil on the spent catalyst surface is a valuable secondary resource [20]. Current methods for recovering residual oil in industrial and laboratory settings include physical separation, solvent extraction, chemical thermal washing, low-temperature pyrolysis, and oxidative roasting. The advantages and disadvantages of each method are summarized in Table 3.

Table 3. Advantages and disadvantages of different pretreatment methods for spent HDS catalysts.

Methods	Instruction	Advantages and Disadvantages
Physical Separation	Deoiling by gravity, cyclone, centrifugal force, etc.	Environmentally friendly; only remove physical oil contaminants; the equipment is expensive.
Oxidative Roasting	Deoiling by oxidation at high temperature and converting metal sulfides to soluble substances.	Removal of oil and carbon completely; high temperature is easy to oxidize the metal or change the structure of the carrier.
Solvent Extraction	Remove oil with organic solvents such as naphtha, acetone, toluene, kerosene and p-xylene.	Mild process, low corrosiveness and recyclable solvent; generates a large amount of waste liquid.
Chemical Thermal Washing	The alkaline solution is combined with surfactant to remove oil by chemical reaction.	Simple process; high cost, producing a large amount of waste liquid.
Pyrolysis	In inert or reducing atmosphere, high-temperature heating decomposes oil stains.	The pyrolysis oil can be recycled, and the treated spent catalyst particles have better dispersion; higher energy consumption.

Physical separation methods separate catalysts from residual oil by means of cyclone action, centrifugal force, density difference and other. This process is simple, does not require chemicals, and avoids secondary pollution. Fu et al. [35] developed cyclone air-stripping deoiling technology to treat spent catalysts, and its technical principle is illustrated in Figure 2. Under a high-temperature gas flow, the catalyst particles are conveyed into a cyclone separator, where high-speed rotation of particles intensifies cyclone deoiling. Meanwhile, the dried catalysts are directly transferred to a gas flow acceleration separator for further treatment. This technology reduces the oil content of spent catalysts from 31.8% to 1.7%, achieving an extremely high deoiling efficiency of 96.7%. Xu et al. [36] investigated the influence of shear stress on the deoiling performance of catalysts in hydrocyclones. Their study demonstrated that higher shear stress accelerates the deoiling process, resulting in a higher deoiling rate and lower energy consumption than traditional mechanical methods, with a deoiling rate reaching 97%. In addition, ultrasonic technology represents another highly efficient and environmentally friendly method for oil removal. It uses the cavitation effect generated by ultrasonic waves to enhance mass transfer and diffusion, thus accelerating oil removal [37]. Yang et al. [38] found that ultrasonic treatment reduces the particle size of spent catalysts, facilitating the release of deoiling. The oil fractions attached to catalyst surfaces can be easily removed with water washing, attaining a deoiling efficiency of 96%. Although modern physical separation methods are more efficient and gentler on the material than traditional methods, their high capital and operating costs have hindered their widespread application.

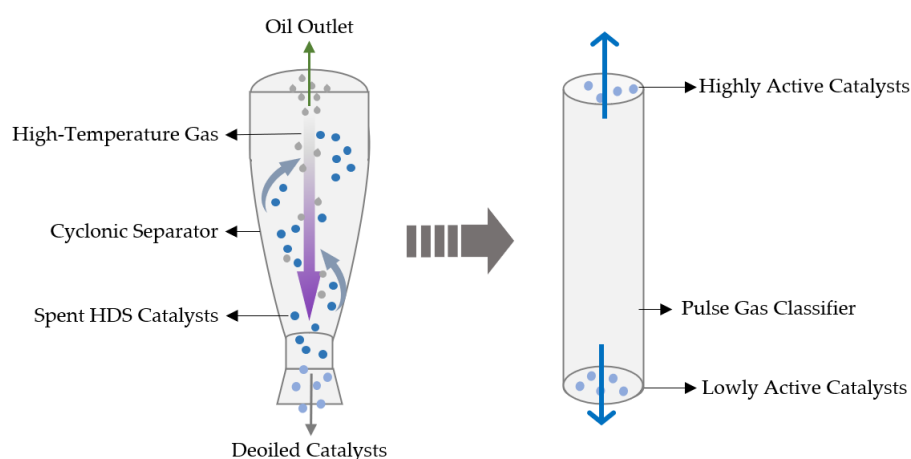


Figure 2. Technical principles of cyclonic gas stripping deoiling and gas flow acceleration classification.

Oxidation roasting is a traditional deoiling process widely used in the pretreatment of spent HDS catalysts. The spent catalyst is roasted at high temperatures in air, whereby residual oil and carbon deposits are oxidized and removed, and insoluble metal sulfides are simultaneously converted into soluble oxides [32,39]. This enhances the efficiency of subsequent hydrometallurgical leaching. The mechanism of oxidation roasting is shown in Figure 3. Inside the deactivated catalyst pores, residual oil, carbon deposits, and sulfides of Mo, Ni, and V accumulate. During oxidation roasting, the carbon deposits are eliminated as CO_2 and CO ; the residual oil decomposes into CO_2 and water vapor; and the metal sulfides are converted into MoO_3 , NiO , and V_2O_5 , releasing SO_2 . This method thoroughly removes oil and clears the catalyst pore channels. However, directly roasting oil-laden catalysts at high temperature causes intense combustion of residual oil, which can easily induce localized overheating. Such overheating accelerates the sintering of metal species into stable, hardly soluble complex spinel phases and damages the support structure, ultimately hindering metal recovery [40,41].

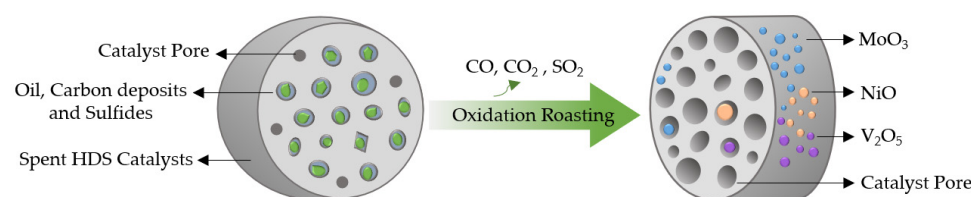


Figure 3. Oxidation roasting process mechanism.

Solvent extraction is based on the principle that “like dissolves like”, dissolving the residual oil from the spent catalyst surface into an organic solvent. Commonly used solvents include toluene, p-xylene, acetone, naphtha, and kerosene [42]. The residual oil on the spent catalyst surface consists predominantly of nonpolar components. Zaheer et al. [13] conducted deoiling experiments using five organic solvents with different levels of polarity. They found that low-polarity solvents, such as kerosene and p-xylene, performed best, achieving oil removal efficiencies of 94% and 96%, respectively. After deoiling, the carbon content of the spent catalyst decreased significantly, while the content of Al, Ni, O, and other metal elements increased. The organic solvent extraction method is relatively mild and preserves the catalyst support structure completely.

Chemical thermal washing combines an alkaline solution with a surfactant to remove oil by altering the interfacial properties between the oil and the catalyst through mechanisms such as emulsification and solubilization [43]. Yang et al. [44] employed a mixture of 0.1 wt.% sodium dodecyl sulfate (SDS) and 0.2 wt.% NaOH to remove residual oil from spent HDS catalysts. Under optimized conditions, the oil removal efficiency reached 99.3%. Comparing catalyst properties before and after deoiling revealed a surface transition from hydrophobic to hydrophilic, which allowed the subsequent calcination temperature to be more controllable. The deoiling treatment substantially improved the metal extraction efficiency of the spent HDS catalyst.

The pyrolysis directly converts residual oil into recoverable pyrolysis oil under high-temperature inert gas conditions, while simultaneously producing by products such as gas and carbon. The effectiveness of pyrolysis depends on the catalysts employed, including metals, metal compounds, molecular sieves, and biomass [45]. Hong et al. [46] systematically compared three deoiling methods: solvent extraction, chemical thermal washing, and pyrolysis. All three methods achieved oil removal efficiencies exceeding 94%. However, whereas solvent extraction and chemical thermal washing generated large volumes of waste liquid, pyrolysis offered higher deoiling efficiency and proved more environmentally friendly. After deoiling, the metal species on the Al_2O_3 support of the spent HDS catalyst are clearly exposed. Unlike the oxidative roasting method, using an inert gas atmosphere during the deoiling process prevents deflagration, protects the spent catalyst support structure, and prevents metal sulfides from transforming into the spinel phase. This provides favorable conditions for extracting metal resources.

3.2. Leaching of Valuable Metals

The valuable metal elements in spent HDS catalysts include Mo, Ni, V, Al, and Co. The extraction processes employed vary for different metals. Mainstream industry extraction methods include acid leaching, alkali leaching, combined acid–alkali leaching, and combined roasting–leaching processes. The main chemical reaction principles governing the extraction of each element are summarized in Table 4.

Table 4. Main chemical reactions for leaching of various elements.

Elements	Chemical Reaction Mechanism	References
Mo	$\text{MoS}_2 + 4\text{H}^+ \rightarrow \text{Mo}_4^{+} + 2\text{H}_2\text{S}$	[24,27,32,47,48]
	$\text{MoS}_2 + 9\text{H}_2\text{O}_2 \rightarrow \text{MoO}_2^{2+} + 2\text{SO}_4^{2-} + 2\text{H}^+ + 8\text{H}_2\text{O}$	
	$\text{MoS}_2 + 3\text{O}_2 \rightarrow \text{MoO}_2 + 2\text{SO}_2(\text{g})$	
	$\text{MoS}_2 + 3.5\text{O}_2 \rightarrow \text{MoO}_3 + 2\text{SO}_2(\text{g})$	
	$\text{Mo}_7\text{S}_8 + 17\text{O}_2 \rightarrow 7\text{MoO}_3 + 8\text{SO}_2(\text{g})$	
	$\text{MoS}_2 + 6\text{MoO}_3 \rightarrow 7\text{MoO}_2 + 2\text{SO}_2(\text{g})$	
	$\text{MoS}_2 + 3.5\text{O}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{MoO}_2\text{SO}_4 + 2\text{SO}_2(\text{g}) + \text{H}_2\text{O}$	
	$\text{MoO}_3 + \text{H}_2\text{SO}_4 \rightarrow \text{MoO}_2\text{SO}_4 + \text{H}_2\text{O}$	
	$\text{MoO}_3 + 6\text{HCl} \rightarrow \text{MoCl}_3 + 3\text{H}_2\text{O}$	
	$\text{MoO}_3 + 2\text{HCl} \rightarrow \text{MoO}_2\text{Cl}_2 + \text{H}_2\text{O}$	
	$\text{MoO}_3 + 2\text{NaOH} \rightarrow \text{Na}_2\text{MoO}_4 + \text{H}_2\text{O}$	
Ni	$\text{NiS} + 4\text{H}_2\text{O}_2 \rightarrow \text{Ni}^{2+} + \text{SO}_4^{2-} + 4\text{H}_2\text{O}$	[24,32,48]
	$\text{NiS} + 1.5\text{O}_2 \rightarrow \text{NiO} + \text{SO}_2(\text{g})$	
	$\text{NiO} + 2\text{HCl} \rightarrow \text{NiCl}_2 + \text{H}_2\text{O}$	
V	$\text{V}_2\text{S}_3 + 5.5\text{O}_2 \rightarrow \text{V}_2\text{O}_5 + 3\text{SO}_2(\text{g})$	[32,33,47,48]
	$4\text{V}_3\text{S}_4 + 31\text{O}_2 \rightarrow 6\text{V}_2\text{O}_5 + 16\text{SO}_2(\text{g})$	
	$\text{V}_2\text{O}_5 + \text{Na}_2\text{CO}_3 \rightarrow 2\text{NaVO}_3 + \text{CO}_2(\text{g})$	
	$\text{V}_2\text{O}_5 + 2\text{HCl} \rightarrow 2\text{VO}_2\text{Cl} + \text{H}_2\text{O}$	
	$\text{V}_2\text{O}_5 + 2\text{NaOH} \rightarrow 2\text{NaVO}_3 + \text{H}_2\text{O}$	
Co	$\text{Co}_3\text{S}_4 + 15\text{H}_2\text{O}_2 \rightarrow 3\text{Co}^{2+} + 4\text{SO}_4^{2-} + 2\text{H}^+ + 14\text{H}_2\text{O}$	[24,49]
	$\text{CoO} + 2\text{HCl} \rightarrow \text{CoCl}_2 + \text{H}_2\text{O}$	
Al	$\text{Al}_2\text{O}_3 + \text{Na}_2\text{CO}_3 \rightarrow 2\text{NaAlO}_2 + \text{CO}_2$	[32,47,48]
	$\text{Al}_2\text{O}_3 + 6\text{HCl} \rightarrow 2\text{AlCl}_3 + 3\text{H}_2\text{O}$	
	$\text{Al}_2\text{O}_3 + 2\text{NaOH} \rightarrow 2\text{NaAlO}_2 + \text{H}_2\text{O}$	

3.2.1. Leaching of Mo

Regarding molybdenum extraction, Marafi et al. [50] used sulfuric acid and citric acid as leaching agents. With ultrasonic assistance, both acids leached Mo with an efficiency of approximately 95%. However, this method lacked selectivity for Mo, and the co-leaching of Mo, Ni, and V complicated the subsequent separation. Gao et al. [51] exploited the complexing ability of oxalic acid with various metals to selectively leach Mo and V, and used NaOH solution to leach Ni from the acidic filter residue, thereby achieving stepwise metal separation. Nevertheless, this method still did not achieve selective leaching of Mo. Compared with acid leaching, alkaline leaching offers greater selectivity toward Mo and is less corrosive to equipment. Huang et al. used caustic soda autoclaving with a low NaOH/Mo ratio to leach Mo and Al, then used Ba(OH)₂ to selectively precipitate Mo [52]. A Mo recovery of 96% was achieved, with Ni remaining in the residue and only 2% of Al dissolving. Zhao et al. [53] leached spent HDS catalysts with a 4.5 mol/L ammonia solution at 413.15 K for 2 h; the Mo leaching efficiency was 96.44%, whereas the V leaching efficiency was only 26%.

To further improve metal recovery, researchers developed combined pyrometallurgical roasting–hydrometallurgical leaching processes, which have been widely applied for Mo extraction from spent HDS catalysts. Such processes integrate pyrochemical pretreatment with hydrometallurgical leaching, using methods such as blank roasting and sodium thermal reduction to convert complex sulfides into easily soluble oxides or oxysalts. Simultaneously, contaminants such as oil, carbon, and sulfur are removed and catalyst pores are cleared, facilitating subsequent leaching with water, acidic, or alkaline solutions [25,32,54]. Chen et al. [55] combined Na₂CO₃ roasting with water leaching to extract metals from spent HDS catalysts. After roasting at 750 °C for 1.5 h and leaching at 65 °C with S/L = 1:6, they achieved Mo leaching efficiency of 98.84%. As shown in Figure 4, Yu et al. [56] subjected

the spent catalyst to oxygen-enriched roasting at 650 °C for 2 h, followed by leaching with 1.5 mol/L oxalic acid, successfully recovering 97.97% of Mo from the spent HDS catalyst. This method enabled the separation of Mo and Ni, reduced high-salinity waste liquid discharge, and allowed the leaching agent to be recycled. Thus, it provides a reliable approach for the green and efficient extraction of Mo. Xu et al. [57] developed a short-process leaching method comprising microwave roasting at 586.5 °C for 31 min, followed by ammonia leaching at 70.2 °C with S/L = 1:10 for 30 min, achieving average leaching efficiencies of 93.88% for Mo, 90.11% for Ni, and 93.28% for V. Furthermore, external field intensification methods, such as ultrasonic, microwave, and mechanical activation can improve leaching kinetics [30]. In Marafi et al.'s study [50], ultrasonic-assisted leaching significantly outperformed conventional stirring. In Adavodi's study [58], compared to agitation leaching, mechanical activation significantly improved the NaOH leaching process. After ball milling for 60 min, 2 mol L⁻¹ NaOH, S/L = 5%, the Mo recovery rate increased from 89% to 94.1%.

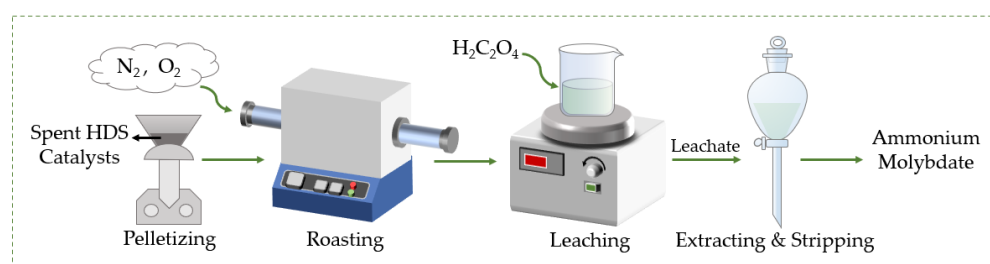


Figure 4. Flow chart of combined process of fire roasting and wet leaching.

3.2.2. Leaching of Ni

The leaching behavior of nickel varies significantly between acidic and alkaline systems. As shown in Table 2, the main Ni phases in spent HDS catalysts are Ni₃S₄, NiS and NiV₃S₆. In strongly acidic media, Ni compounds dissociate into Ni²⁺ ions that readily enter the solution, yielding high leaching rates. Marafi et al. [50] reported Ni leaching rates exceeding 95% when using 10% H₂SO₄ and 10% citric acid. Ilhan found that [59] a sample roasted at 500 °C was used for dissolution experiments. Under optimal dissolution conditions, the leaching efficiency of Ni with oxalic acid solution reached only 19%, while the dissolution efficiency of Mo was 92%. These results suggest that the acid dissociation of Ni compounds is not the only factor that governs extraction. Rather, leaching efficiency is determined by the acid's ability to complex with Ni²⁺ and the resulting complexes' solubility. Nickel sulfate and nickel citrate are highly soluble in water, whereas nickel oxalate is virtually insoluble in both water and acids, severely hindering Ni leaching [60]. The leaching efficiency of Ni also depends strongly on the pH of the solvent. Unlike Mo, Ni dissolves readily in strongly acidic solutions, but its solubility declines as the pH increases. In alkaline media such as NaOH or Na₂CO₃ solutions, Ni leaching is suppressed and nearly all of the Ni remains in the solid residue. The study by Huang et al. [52] cited above provides evidence for this.

3.2.3. Leaching of V

Vanadium is a multivalent element whose valence state, physicochemical properties, and coordination behavior vary markedly under different redox conditions. Common vanadium phases found in spent HDS catalysts include V₂S₃, V₃S₄, complex Ni-V-S solid solutions, and minor amounts of vanadium oxide. Low-valent vanadium sulfides are stable and highly chemically inert at ambient temperature and pressure. V(III) and V(II) species are difficult to convert into hydrated ions or soluble hydroxyl complexes, nor can they readily form vanadates [61,62]. Consequently, vanadium cannot be effectively leached

using conventional acids, alkalis, or ammonia solutions. In Zhao's study [53], the leaching efficiency of V using ammonia water was only 27%. To overcome the limitations of single-solution leaching, a combined acid–alkaline leaching process has been developed. An alkaline solution selectively leaches Mo and V, while an acidic solution oxidizes low-valent V to readily soluble high-valent species and simultaneously decomposes insoluble sulfides in the spent leachate. Zhao et al. [53] introduced H_2O_2 into the ammonia leaching system. Under optimal conditions, as the H_2O_2 concentration increased from 0 to 2.5 mol/L, V(IV) was oxidized to V(V) and the vanadium extraction efficiency increased from 26.54% to 39.73%. Chen et al. [63] used 300 g/L of $(\text{NH}_4)_2\text{SO}_4$ and 1.5 mol/L of H_2O_2 to extract V, Mo, and Ni from spent HDS catalysts. At 55 °C, pH = 3.5, and S/L = 15:1, the maximum V leaching efficiency was 92%. Although easy to operate, this method consumes large amounts of oxidant and generates substantial waste liquid, hindering large-scale industrial application. More researchers have therefore adopted the combined roasting–leaching process to enhance V leaching. Ying et al. [33] used roasting–HCl leaching to extract valuable metals; with 5 mol/L HCl, 99.3% of the V was leached, although Al, Mo, and Ni were co-extracted simultaneously. Wang et al. [32] performed roasting at 650 °C and used 35% NaOH to extract approximately 97% of the V, while the alkaline leaching step also achieved the selective separation of Mo and V from Al_2O_3 .

3.2.4. Leaching of Co

Cobalt is present in spent HDS catalysts at much lower concentrations than molybdenum and nickel, and studies specifically targeting Co recovery remain scarce. Co exhibits a leaching behavior similar to Ni. It dissolves readily under strongly acidic conditions, whereas under alkaline conditions nearly all of the Co is retained in the residue. After oxidation treatment, cobalt oxides become readily leachable. However, excessively high roasting temperatures can promote the conversion of cobalt into the refractory CoAl_2O_4 spinel phase [49,64]. Banda et al. [65] leached spent HDS catalysts with 3 mol/L hydrochloric acid at 90 °C and S/L = 5% for 60 min, achieving dissolution efficiencies of 97% for Mo and 94% for Co. Arslanoğlu et al. [24] employed oxidative roasting combined with $\text{Na}_2\text{S}_2\text{O}_8$ to enhance metal extraction from spent HDS catalysts. Under this combined treatment, Co was fully oxidized, achieving a leaching rate of 98.5%. However, Mo, Ni, and Al are co-dissolved in the leachate.

3.2.5. Leaching of Al

Spent HDS catalysts are supported on Al_2O_3 and therefore contain a high proportion of aluminum, primarily in the form of $\gamma\text{-Al}_2\text{O}_3$. However, during prolonged operation, part of the $\gamma\text{-Al}_2\text{O}_3$ reacts with metal components and is progressively converted into insoluble spinel phases, such as NiAl_2O_4 and CoAl_2O_4 , increasing the difficulty of aluminum recovery. Strong acid leaching simultaneously dissolves Al, Mo, Ni, V, and Co, resulting in high metal extraction rates. However, high Al^{3+} concentrations together with other metal ions complicate subsequent separation and raise treatment costs [27]. In contrast, alkaline leaching processes exploit the amphoteric nature of Al_2O_3 by using NaOH or Na_2CO_3 to selectively dissolve Al, Mo, and V, thereby achieving a preliminary separation of the metal elements.

3.2.6. Comparison of Element Extraction Methods

Table 5 summarizes the main extraction methods for valuable elements in spent HDS catalysts, as reported in the cited literature. Single-stage acid leaching can leach large quantities of Mo, Ni, V, Co, and Al, but co-dissolution of multiple metals increases the burden of subsequent separation, and lower S/L results in a large volume of spent liquor. In contrast, single-stage alkaline leaching selectively extracts Mo while nearly completely

preventing the leaching of Al, enabling the separation of Mo and V from the Al_2O_3 support. This process avoids extensive co-dissolution and reduces the difficulty of separating other elements downstream. Roasting–leaching processes enhance the efficiency of metal leaching, with Mo extraction rates exceeding 96%. However, such processes intensify the co-dissolution of multiple metals in strong acid and strongly oxidizing salt leaching systems. High-temperature roasting increases energy consumption and generates SO_2 , adding to the cost of off-gas treatment. Conversely, higher S/L reduces water consumption during leaching and decreases the volume of spent liquor. H_2O_2 -assisted leaching processes can achieve high metal extraction rates without high-temperature roasting, but they are costly, and the technology is still immature, limited to laboratory-scale research.

Table 5. Major methods for extracting valuable elements.

No.	Method	Major Metals in the Raw Materials	Reagents	Experimental Conditions	Extraction Rate	References
1	Acid Leaching	Mo, Ni, V, Al	10 wt% H_2SO_4 / 10 wt% Citric acid	Ultrasound-Assisted Leaching, S/L = 1:40, 60 °C, 6 h	Mo: 95.7%, Ni: 95.7%, V: 94.8%, Al: 18.1	[50]
2		Mo, Ni, V, Al	1 mol/L Oxalic acid	S/L = 1:20, 60 °C, 2 h	Mo: 93.07%, Ni: 74.21%, V: 86.64%	[51]
3		Mo, Ni, Co, Al	3 mol/L HCl	S/L = 1:20, 90 °C, 1 h	Mo: 97%, Ni: 33.8%, Co: 94%, Al: 84.5%	[65]
4	Alkali Leaching	Mo, Ni, Al	3 mol/L NaOH	NaOH:2Mo = 1.2, S/L = 1:4, 160 °C, 2 h	Mo: 96%, Al < 0.2%	[52]
5		Mo, V, Ti, Al	4.5 mol/L $\text{NH}_3 \cdot \text{H}_2\text{O}$	S/L = 1:20, 200 °C, 2 h	Mo: 96.44%, V: 26%	[53]
6		Mo, Ni, V, Co, Al	2.5 mol/L NaOH	S/L = 1:20, 60 °C, 1 h	Mo: 89%, V: 77.7%	[58]
7		Mo, Ni, V, Co, Al	2 mol/L NaOH	Ball Milling 60 min, S/L = 1:20, 60 °C, 1 h	Mo: 94.1%, V: 97.4%	[58]
8	Roasting— Water Leaching	Mo, Ni, V, Al	Na_2CO_3	Roasting: 750 °C, 1.5 h, Catalyst: Na_2CO_3 = 1:1, Leaching: S/L = 1:6, 65 °C	Mo: 98.84%, V: 99.22%, Al: 41.67%	[55]
9	Roasting— Acid Leaching	Mo, Ni, Al	1.5mol/L Oxalic acid, 1 mol/L Acetic acid	Roasting: 650 °C, 2 h, Leaching Mo: S/L = 1:10, 50 °C, 2 h, Leaching Ni: 20 °C, 2 h	Mo: 97.97%, Ni: 1.3%, Ni: 96.77%	[56]
10		Mo, Ni, Al	1 mol/L Oxalic acid	Roasting: 500 °C, 4 h, Leaching: S/L = 1:100, 40 °C, 3 h	Mo: 92%, Ni: 19%, Al: 28%	[59]
11		Mo, Ni, V, Al	5 mol/L HCl	Roasting: 500 °C, 2.5 h, Leaching: S/L = 1:8, 85 °C, 3 h	Mo: 96.2%, Ni: 94.2%, V: 99.3%, Al: 96.1%	[33]
12	Roasting— Alkali Leaching	Mo, Ni, V, Al	35% NaOH	Roasting: 650 °C, Leaching: S/L = 1:10, 110 °C, 5 h	Mo: 96%, V: 97%, Al: 88%	[32]
13		Mo, Ni, V, Al	30% NaOH	Roasting: 1000 °C, Leaching: S/L = 1:8, 110 °C, 5 h	Mo: 96%, V: 91%	[32]
14	Microwave Calcination— Ammonia Leaching	Mo, Ni, V, Al	6 mol/L $\text{NH}_3 \cdot \text{H}_2\text{O}$	Roasting: 586.5 °C, 31 min, Leaching: S/L = 1:10, pH = 12, 70.2 °C, 30 min	Mo: 93.88%, Ni: 90.11%, V: 93.28%	[57]
15	Roasting— Salt Leaching	Mo, Ni, Co, Al	0.4 mol/L $\text{Na}_2\text{S}_2\text{O}_8$,	Roasting: 500 °C, 2 h, Leaching: S/L = 1:12.5, 50 °C, 1.5 h	Mo: 92.6%, Ni: 95.3%, Co: 98.5%, Al: 21.3%	[24]

Table 5. Cont.

No.	Method	Major Metals in the Raw Materials	Reagents	Experimental Conditions	Extraction Rate	References
16	H ₂ O ₂ —Ammonia Leaching	Mo, V, Ti, Al	1 mol/L H ₂ O ₂ , 4.5 mol/L NH ₃ ·H ₂ O	S/L = 1:20, 140 °C, 2 h	Mo: 95.13%, V: 46.25%	[53]
17	H ₂ O ₂ —Salt Leaching	Mo, Ni, V, Al	1.5 mol/L H ₂ O ₂ , 300 g/L (NH ₄) ₂ SO ₄	pH = 3.5, S/L = 1:15, 55 °C, 3 h	Mo: 84%, Ni: 97%, V: 92%	[63]

Based on the above data, Mo is the most abundant valuable metal in spent HDS catalysts and has the widest range of extraction routes. Virtually all processes achieve recovery > 92%. For the recovery of only Mo, mechanically activated single-stage alkaline leaching offers relatively favorable performance. Adavodi et al. conducted a semi-quantitative economic assessment of the mechanically activated alkaline leaching process, demonstrating its ability to extract large amounts of Mo in shorter times at lower temperatures. Although the annual electricity consumption is approximately 4.5 USD/t higher than that of conventional stirring, the capital expenditure and reagent consumption are lower. The higher Mo recovery increases the overall revenue by 484 USD/t while reducing environmental pollution, thus presenting a distinct advantage [58]. For full-component recovery of Mo, Ni, V, and Co, the roasting–alkaline leaching process is preferred. Yu et al. used life cycle assessment (LCA) to evaluate the environmental impact of various recovery processes. They found that the roasting–alkaline leaching process has lower energy and water consumption. Its primary energy demand (PED) is 990 MJ/t lower than the hydrometallurgical process. Its water consumption is 60 kg/t lower. Its direct cost is only 65.52% of the hydrometallurgical process’s cost [56].

3.3. Selective Separation of Valuable Metals

Strong acid leaching tends to co-dissolve multiple metals into the leachate. In contrast, alkaline leaching takes advantage of the different solubilities of metal compounds to separate Mo and V from the Al₂O₃ support while retaining Ni and Co in the leaching residue, thereby achieving preliminary metal separation at the source. The targeted extraction of these metals provides an excellent raw material basis for producing high-purity products that meet industrial specifications. Commonly employed separation techniques include solvent extraction, chemical precipitation, and ion exchange [66].

3.3.1. Separation of Aluminum

A high concentration of Al³⁺ in the leaching system can interfere with the selective precipitation of Mo and V. It may also lead to the formation of insoluble compounds that encapsulate other metals, reducing product purity. Therefore, aluminum must be removed from the leachate first. As shown in Table 3, aluminum exists as Al³⁺ in acidic leachates and as AlO₂[−] in alkaline leachates. The separation strategy involves adjusting the pH to precipitate both species as Al(OH)₃. The experimental results of Wiecka et al. [67] showed that when 30% NaOH was used to adjust the pH of the acid leachate, aluminum began to precipitate at pH > 3. However, this approach also caused co-precipitation of Mo and Co, thereby limiting the overall metal recovery. Singh [68] mixed NaOH with the spent catalyst at a 1:1 ratio for roasting, then leached the roasted product with pure water. This process simultaneously dissolved 99% of Al and V. Subsequently, the pH of the leachate was adjusted to 9.5 with HCl to achieve selective precipitation. This yielded an aluminum recovery of 99%, while retaining all vanadium in the filtrate. The resulting Al(OH)₃ product was 98% pure.

3.3.2. Selective Separation of Mo and V

Mo and V frequently coexist in leachates, and chemical precipitation is the most economical method for their separation. Kar et al. [54] adjusted the pH of the alkaline leachate to two with HCl to induce molybdate polymerization, then added ammonia solution dropwise to convert the polymolybdate ions into an ammonium polymolybdate precipitate, achieving approximately 90% Mo recovery. Nguyen et al. [69] extracted Mo from the HCl leachate of spent HDS catalysts using TBP solvent, followed by stripping with HCl. High-purity CaMoO_4 powder was then prepared by adding CaCl_2 at pH = 5 and 90 °C, with recoveries of 96% for Mo. These studies demonstrate that chemical precipitation can achieve the selective separation of Mo and Co, but high levels of impurities in the leachate can affect product purity.

Considerable research has shifted toward solvent extraction, which uses differences in metal ion coordination affinities with various extractants to achieve selective separation. Park et al. [27] obtained a low-acid leachate through sulfuric acid roasting, then water leaching. They sequentially extracted MoO_2^+ and Ni^{2+} using LIX84-I and Cyanex 272, which separated Mo, Ni, and Al. Sun et al. [70] used a mixture of 25% D2EHPA, 5% TBP, and 70% sulfonated kerosene to extract Mo and V. When pH = −0.24, the extraction efficiency for Mo reached 99%; when pH = 1.07, the extraction efficiency for V reached 99%.

For solutions with low Mo concentrations, ion exchange provides a more effective separation. Nguyen et al. [71] used AG1-x8 and a two-stage anion exchange process to separate Mo at pH = 0.5 and V at pH = 1.6, and achieving Mo and V recoveries of 96.2% and 95.7%, respectively. Recently, researchers have developed more efficient and selective molybdenum extraction processes. Huang et al. [72] proposed a cyclic Mo extraction process combining ethanol antisolvent precipitation with acid leaching, in order to crystallize and separate Ni and Al, while 96% of the Mo remains in the mother liquor. The mother liquor is distilled to recover the ethanol. Then, the Mo-rich solution undergoes TBP extraction directly. The residual sulfuric acid can be recycled back to the leaching step. This process requires no pH adjustment, generates no sludge, and enables the reuse of both the organic solvent and sulfuric acid; single-pass Mo recovery exceeds 85%, effectively reducing the environmental impact of conventional hydrometallurgical processes.

3.3.3. Selective Separation of Ni and Co

The selective extraction of Ni and Co is particularly challenging because of their similar chemical properties. At the ionic level, both Ni^{2+} and Co^{2+} have the same charge, so it is difficult to separate them by simple precipitation. Many researchers have turned to solvent extraction for recovering Ni and Mo. Banda et al. [65] extracted Co from the Mo-depleted mother liquor by three-stage countercurrent extraction with Alamine 308, achieving a recovery of 99%. Chen et al. [48] used N235-TBP to extract V, Mo, and Fe from alkaline leachate and subsequently recovered Ni from the extractant residue with HBL 110, attaining Ni recovery as high as 99.92%. In contrast, Arslanoğlu et al. [24] separated Mo, Ni, and Co from an acid leachate by pH-controlled chemical precipitation. The acid leachate was first adjusted to pH two with aqueous ammonia and hydrochloric acid to precipitate ammonium molybdate; the pH was then raised to six, and dimethylglyoxime (DMG) was added to precipitate Ni^{2+} . Finally, the pH was adjusted to 11 to precipitate Co^{2+} . The recovery rates for Mo, Ni, and Co were 89.8%, 81.2%, and 86.5%.

Solvent extraction provides higher metal recovery and product purity, generates less solid waste, and results in a cleaner process overall due to the selective extraction of metal ions. Although industrial-scale production is costly, organic solvents can be reused. Chemical precipitation is simple to operate and inexpensive, but each solid-phase precipitation step may result in the loss of other metals. This leads to incomplete metal recovery and

affects the purity of metal products. Ion exchange can achieve higher enrichment and purity, but it is operationally complex and generates large volumes of regeneration waste. It also has limited resin exchange capacity. Processing high-concentration feed solutions requires frequent regeneration and high-level automation control. Overall, solvent extraction provides the best balance of selectivity, product quality, and process maturity for separating metal ions from complex leachates.

4. Discussion

Spent HDS catalysts contain various strategic metals, making the complete recovery of all valuable components the most advantageous utilization pathway. The full-component resource utilization of spent HDS catalysts can be summarized as the technical route shown in Figure 5. Regarding spent catalyst pretreatment, roasting is easy to operate and can effectively remove impurities such as residual oil and carbon deposits while converting insoluble metal compounds into soluble forms, thereby facilitating subsequent metal leaching and shortening the overall resource recovery process. Ilhan and Valverde Jr. [49,59] examined the effect of particle size on the leaching efficiency of spent HDS catalysts. Their results showed that reducing the particle size did not significantly change the leaching efficiency. Consequently, the crushing and grinding step can be omitted in conventional pretreatment. In the hydrometallurgical leaching unit, acid leaching co-dissolves multiple metals, complicating downstream separation and generating large volumes of acidic waste liquid. Alkaline leaching partially separates metals at the source, thereby simplifying targeted recovery and achieving elemental partitioning, which facilitates the selective separation of metals. When the concentration of Al^{3+} in the leachate is high, aluminum can be removed by precipitation before recovering other metals. Solvent extraction is widely adopted because it is environmentally friendly, efficient, and recyclable. For molybdenum recovery, Mo is loaded into the organic phase as an organic complex and subsequently stripped back into the aqueous phase as MoO_4^{2-} using an alkaline solution. The introduction of NH_4^+ converts MoO_4^{2-} into an ammonium molybdate precipitate. High-temperature roasting volatilizes the ammonia and water vapor, yielding a high-purity MoO_3 product [73,74]. Leszczyńska-Sejda et al. [39] obtained a sodium molybdate solution by oxidative roasting and NaOH leaching, then precipitated Mo with HCl to prepare commercially valuable molybdic acid. In another study, the extracted molybdenum was sulfidized to MoS_2 and used to fabricate a photocatalytic material for hydrogen production via visible-light water splitting [75].

The selective recovery of V follows a process similar to that of Mo: the pH of the mother liquor is adjusted, V is selectively extracted using an organic solvent, and V_2O_5 product is obtained through subsequent stripping, precipitation, and roasting. Nickel and cobalt are enriched in the alkaline leaching residue and in the water leaching residue after sodium salt roasting. Acid leaching of these residues yields a solution containing Ni^{2+} and Co^{2+} . The two metals can be extracted separately at different pH levels and then removed from the organic phases, yielding individual metal solutions. Adding oxalic acid solution precipitates Ni^{2+} and Co^{2+} as their oxalates.

The roasting–hydrometallurgical leaching–solvent extraction process, due to its maturity, high metal recovery rates, and high product purity, enables the efficient recovery of valuable metals from spent HDS catalysts. However, this process still faces issues in industrial applications, such as high energy consumption, large volumes of wastewater discharge, and high costs associated with organic solvents. Researchers have proposed an element extraction method involving antisolvent precipitation followed by acid leaching; however, it has not yet been adapted for large-scale industrial application. Numerous experimental studies have shown that metal extraction can be substantially enhanced and

the leaching time significantly shortened by employing intensification methods such as ultrasound, microwaves, mechanical activation, and use of oxidizing agents, either individually or in combination [76,77], but these methods are not yet suitable for large-scale industrial applications.

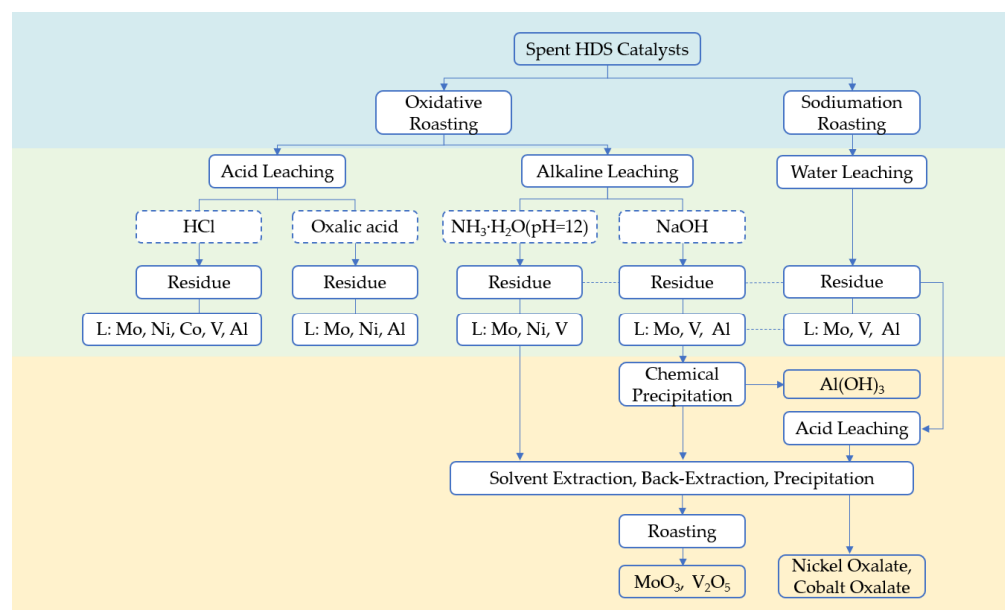


Figure 5. Process flow diagram for the full-component recovery of metals from spent HDS catalysts.

In recent years, researchers have devoted increasing attention to developing deep eutectic solvents and bio-based leaching agents, aiming to establish metal recovery processes with shorter flowsheets and reduced environmental impact. For example, Güloğlu et al. [78] used a choline chloride and p-toluene sulfonic acid deep eutectic solvent to extract Co and Mo with high recovery rates at pH 5.5 and 9.5. Aluminum remained in solution throughout the process. Similarly, Wang [79] developed an extraction process using a deep homogeneous aqueous solvent composed of betaine hydrochloride and p-toluene sulfonic acid, achieving leaching efficiencies exceeding 90% for Ni, Mo, and V while suppressing the Al leaching efficiency to 9.82%.

LCA into the study allows for the evaluation of the environmental impact of the entire process, thereby identifying the main sources of pollution and clarifying directions for process optimization [56]. Furthermore, applying machine learning algorithms to the process allows the simulation of how various parameters affect metal recovery, enabling more precise control of operating conditions, reducing the number of experiments required, and accelerating process optimization. Sureya et al. employed algorithms to identify optimal process parameters for Ni recovery from laterite ore [80]; they achieved a 97.74% Ni recovery, reduced acid consumption by 20%, decreased total costs by 15%, and cut carbon emissions by 134.5 kg CO₂ eq/t. In summary, green leaching systems, such as deep eutectic solvents and bio-based leaching agents, have considerable potential. The introduction of LCA and algorithms provide quantitative tools for environmental assessment and process parameter optimization across the entire flowsheet. Future research could focus on developing more economical and environmentally friendly leaching and extraction systems, advancing intensification techniques, such as microwave, ultrasound, and mechanical activation, from the laboratory to industrial scale, and systematically optimizing the full-component recovery process for spent HDS catalysts with the aid of algorithms and LCA methodology. This would achieve the complete and quantitative utilization of all components while minimizing energy consumption and wastewater discharge.

5. Conclusions

Spent HDS catalysts are an important secondary source of strategic metals, and maximizing the utilization of these valuable resources is of considerable significance. This review summarizes the main technologies and the current state of metal resource recovery from spent HDS catalysts in three stages: pretreatment, metal extraction, and selective separation. An integrated process route for the full-component recovery of valuable metals is outlined by incorporating economic feasibility and environmental assessment. The roasting–hydrometallurgical leaching–solvent extraction approach efficiently extracts metal elements from spent HDS catalysts and produces them as high-purity products. However, extracting these metals in a greener, more cost-effective manner suitable for large-scale industrial application remains a question worthy of further consideration. Future research could focus on developing green, efficient leaching systems; exploring novel technologies for targeted metal resource separation; and optimizing high-value resource recovery processes for spent HDS catalysts by integrating emerging technologies. This would ultimately achieve the full-component recovery and efficient utilization of spent catalysts.

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