PROPOSED METHODS FOR RECOVERING CRITICAL METALS FROM SPENT CATALYSTS

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Summary

The Bureau of Mines is investigating chlorination methods, various hydrometallurgical approaches, and pyrometallurgical roasting procedures to recover Ni, Mo, W, Cu, and Cr from spent hydroprocessing, hydrogenation, and high-temperature shift catalysts. Laboratory work to date has resulted in extraction of 73 to 99% of the metals from these wastes. Total processing schemes for recovering the metals have been proposed. Economic feasibility of the proposed processing schemes is being evaluated.

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Introduction

Two objectives of the Bureau of Mines are to help assure an adequate supply of critical metals for the United States to meet economic and strategic needs and to help reduce or avoid dependence on foreign supplies. Attaining these objectives necessitates evaluation of the potential recovery of metals from secondary sources as well as from domestic mineral resources. Spent hydroprocessing, hydrogenation, and high-temperature shift catalysts were identified in a Bureau contract study (1) as a potential source of Ni, Co, Mo, W, V, Cr, Cu, and Zn. The study revealed that more than 12 million kilograms per year of those metals were discharged in spent catalysts and that less than half of the metals were recovered or recycled.

Representative patent literature on processing approaches for recovering metals from spent catalysts was reported in the Bureau contract study. A few papers on the subject were found in journal publications. In general, chlorination (2), pressure leaching with ammonium hydroxide-ammonium carbonate (3, 4) or with sodium hydroxide (3), atmospheric leaching with sodium hydroxide (5), and sodium carbonate roasting (6) were applied. Most processing approaches described in the literature were aimed at recovering molybdenum or molybdenum, vanadium, and tungsten, but not at recovering nickel and cobalt. In many cases, metal extraction was low and metal separation was not complete. The chlorination method described in the literature to selectively absorb aluminum chloride with sodium chloride and molybdenum chloride with potassium chloride was tried in our laboratory and found to be not selective. In 1983, the Bureau began characterization studies on spent, nonprecious metal containing catalysts and began evaluation of a variety of approaches to identify suitable total processing schemes for recovering all of the critical metals present. Initial research results that emphasized spent catalyst characterization and metal extraction were reported at the 1985 Annual AIME Meeting in New York (7).

The Bureau's approaches can be divided generally into three categories: (a) chlorination, (b) leaching with ammonium hydroxide-ammonium sulfate, sodium hydroxide, and sulfuric acid, and (c) roasting with sodium carbonate followed by water leaching. Chlorination of metal oxide requires a reductant such as carbon or carbon monoxide. Hydroprocessing spent catalysts contain over 30% of alumina. One purpose of the research is to recover critical metals not aluminum. If alumina is chlorinated during chlorination of spent catalyst, the chlorination will consume a lot of chlorine. Chlorination of alumina can be impeded by controlling the CO_2/CO ratio as shown in the following equation: $Al_2O_3 + 3CO + 3Cl_2 \rightarrow 2AlCl_3 + 3CO_2$. This was accomplished by utilizing natural carbon in the spent catalyst and adding air to chlorine and nitrogen, or utilitizing carbon monoxide; but the addition of air did not impede chlorination of nickel oxide and molybdenum oxide significantly (7). Recently, it also was found that chlorination of tungsten oxide was impeded significantly by controlling the CO_2/CO ratio, or by adding air to chlorine and nitrogen and the carbon to the spent catalyst, in the case of insufficient natural carbon content in the catalyst.

Vapor pressure-temperature relationships of the metal chlorides (8, 9, 10, and 11) pertinent to spent catalyst chlorination are shown in Figure 1. High vapor pressure of $MoCl_5$, WCl_6, or $AlCl_3$ at an elevated temperature and low vapor pressure of $NiCl_2$ indicate that the former would volatilize, while the latter would remain in the spent charge during chlorination. The $NiCl_2$ and $CuCl_2$, or $FeCl_2$ and $CrCl_3$ in a reducing atmosphere, or $FeCl_3$ and CrO_2Cl_2 in an oxidizing atmosphere, have similar vapor pressures. These chlorides would be difficult to separate by volatilization; therefore, a lesser effort was placed on recovering metals by chlorinating spent catalysts containing these metal combinations.

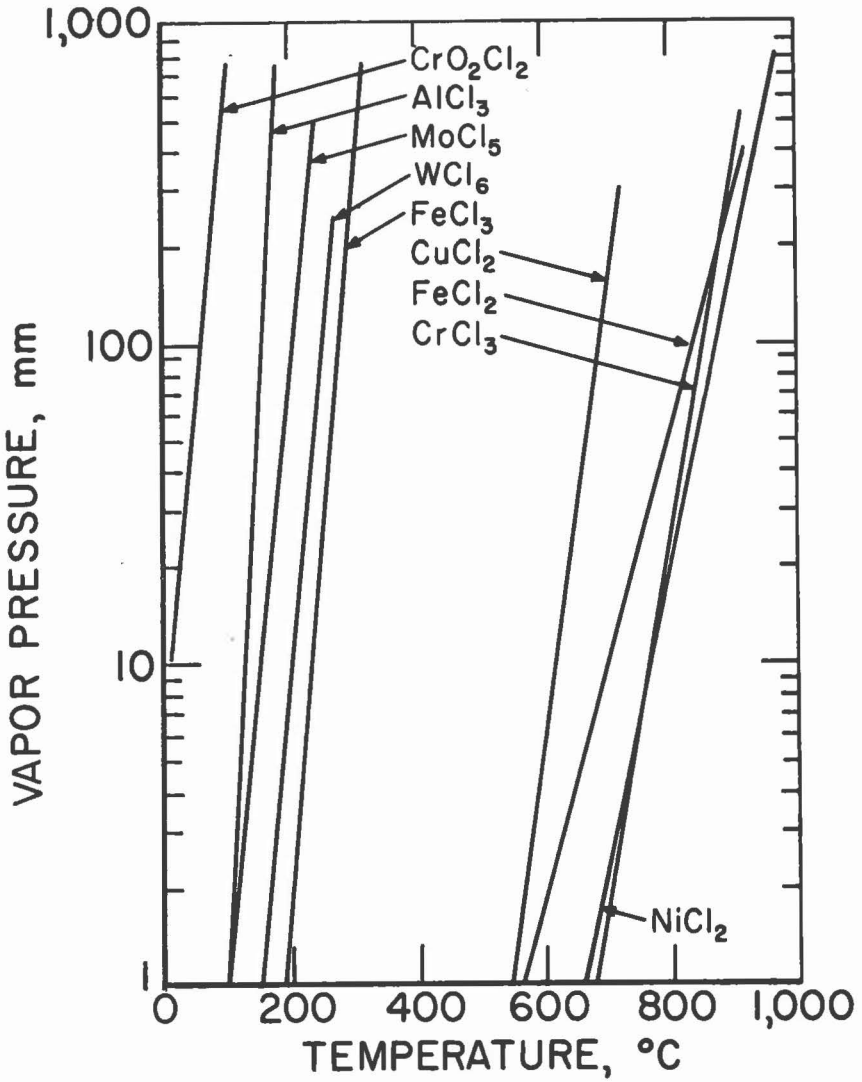

FIGURE 1 - Vapor pressure of metal chlorides pertinent to spent catalyst chlorination.

Initial studies on leaching approaches with ammonium hydroxide-ammonium sulfate or ammonium hydroxide-ammonium carbonate showed that both approaches resulted in about the same metal extraction. It also was found that pressure leaching in an oxygen atmosphere with ammonium hydroxide-ammonium sulfate, sodium hydroxide, or sulfuric acid for most spent catalysts was not necessary.

This paper presents the results to date of metal extraction, metal separation, and proposed total processing schemes for recovering critical metals from Ni-Mo-Al, Ni-W-Al, Ni-Cu-Si, and Cr-Fe spent catalysts. Results of research on spent catalysts that contain cobalt or vanadium will be published later in a Bureau report.

Materials

Representative spent catalysts were supplied by INCO Research and Development Center as part of a Bureau contract (1). Chemical analysis data and phases identified by X-ray diffraction are shown in Table I.

TABLE I. - Metal, carbon, sulfur, and water contents in spent catalysts

Spent catalyst	Metal, %				Nonmetal, %			X-ray identified phases
Hydroprocessing:	Ni	Mo	W	Al	C	S	H_2O	
Ni-Mo-Al (sample A).	1.7	8.9	NAp	29.6	6.7	5.6	8.5	Poorly crystallized Al_2O_3
Ni-Mo-Al (sample B).	2.0	10.1	NAp	31.2	1.6	.4	8.3	Poorly crystallized Al_2O_3
Ni-W-Al.....	3.4	NAp	13.1	33.8	1.0	.3	5.0	Poorly crystallized Al_2O_3
Hydrogenation:	Ni	Cu	Si		C	S	H_2O	
Ni-Cu-Si.....	12.7	4.9	28.3		0.2	0.04	0.4	Alpha quartz, crystobalite, tridymite, NiO, and/or $Cu_{0.2}Ni_{0.8}O$
High-temperature shift	Cr	Fe			C	S	H_2O	
Cr-Fe.....	5.4	61.3			1.8	0.07	0.3	$(Fe,Cr)_3O_4$

NAp Not applicable.

Commercial-grade ammonium hydroxide and ammonium sulfate and reagent-grade chlorine, sodium hydroxide, sodium carbonate, and sulfuric acid were used for metal extraction studies.

Four types of commercially available extractants were selected for initial metal separation studies as follows: (a) chelating type - LIX 64N[3], LIX 63, and Kelex 100; (b) ionic type - Alamine 304 and Aliquat 336; (c) acidic type - DNNSA (dinonylnaphthalene sulfonic acid), naphthenic acid, and D2EHPA (di-2 ethylhexyl phosphoric acid); and (d) solvating type - TBP (tributyl phosphate). In some cases, a mixture of these extractants was used. Kerosene or naphtha was used as a diluent, and isodecanol was used as a modifier in some cases.

Experimental Work

Chlorination was carried out in a 1-1/4- by 48-in Vycor tube reactor. The reactor was equipped with a condenser and a solids collector. A 50-g catalyst charge was used for the chlorination of the Ni-Mo-Al or the Ni-Cu-Si catalyst, and a 25-g catalyst charge was used for the Ni-W-Al catalyst. The Ni-W-Al catalyst has a bulk density of 0.66 g/cm^3, and Ni-Mo-Al has a bulk density of 1.06 g/cm^3. As a prechlorination step most of the catalyst materials was roasted at 400° C for 30 min with 400 mL/m N_2 to remove the sulfur and moisture (up to 5% each) prior to the chlorination.

[3]Reference to specific brands is made for identification only and does not imply endorsement by the Bureau of Mines.

Spent catalysts were then chlorinated at 450° C for 30 min with Cl_2 and air, or Cl_2 and CO. The catalyst bed was fluidized during chlorination with N_2. Initially, the flow rate settings of Cl_2, air, or CO were varied between 30 and 100 mL/min, and N_2 was varied between 200 and 900 mL/min. Processing conditions representing the best chlorination results used in these tests are shown in Table II.

TABLE II. - Metal extraction from various catalysts by chlorination

Spent	Flow rate, mL/min				Extraction, %				
catalyst	Cl_2	Air	CO	N_2	Ni	Mo	W	Cu	Al
Ni-Mo-Al....	60	60	NAp	650	83	89	NAp	NAp	4
Ni-W-Al.....	50	NAp	50	900	73	NAp	82	NAp	5
Ni-Cu-Si....	100	NAp	100	200	88	NAp	NAp	84	NAp

NAp Not applicable.

Leaching was carried out in a 2-L Pyrex glass reaction flask equipped with a stirrer, a reflux condenser, and a thermometer. A 50- or 100- catalyst charge was used for leaching experiments. The spent catalyst was leached with 1 L of 100 g/L NH_4OH plus 300 g/L $(NH_4)_2SO_4$ at 80° C for 2 h, or 1 L of 10 or 20 g/L NaOH at 100° C for 2 h, or 1 L of 50 or 100 g/L H_2SO_4 at 100° C for 1 h.

For the sodium carbonate roasting followed by water leaching approach, one part sodium carbonate was roasted with four parts spent catalysts at 600° C for 2 h in air. The roasted catalyst was then leached in water at 100° C for 2 h.

Metal ions in leach soluton were separated either by solvent extraction or by precipitation. For solvent extraction, the aqueous solution and solvent were contacted by vigorous shaking in a separator funnel on a wrist-action shaker for 30 min at room temperature. An organic to aqueous ratio of 1:1 was used.

Results and Discussions

With the chlorination approach more than 80% of the Ni, Mo, W, and Cu was extracted from most of the spent catalyst materials evaluated; however, only 73% of the Ni was extracted from the Ni-W-Al spent catalyst. Results are shown in Table II. Aluminum extraction was below 5%. For the Ni-Mo-Al catalyst material, chlorination produced two volatile products, a major phase of MoO_2Cl_2 and a minor phase of $AlCl_3$. Less than 5% of the $AlCl_3$ transferred to the condenser, and the rest of the $AlCl_3$ remained with the $NiCl_2$ in the chlorinated charge. For the Ni-W-Al catalyst, the chlorination produced two volatile products, a major phase of WCl_6 and a minor phase of $AlCl_3$. Less than 20% of the $AlCl_3$ transferred to the condenser, and the rest of the $AlCl_3$ remained with the $NiCl_2$ in the chlorinated charge. The MoO_2Cl_2 or WCl_6 was dechlorinated with air to produce MoO_3 or WO_3. Alternatively, the MoO_2Cl_2 or WCl_6 was hydrolyzed in water to produce MoO_3 or WO_3. For the Ni-Cu-Si catalyst, most of the $NiCl_2$ and $CuCl_2$ stayed in the chlorinated charge.

Results of the leaching approaches are shown in Table III. An ammnium hydroxide-ammonium sulfate leaching approach selectively extracted Ni and Mo from the Ni-Mo-Al catalyst but did not effectively extract metals from the Ni-W-Al or Ni-Cu-Si catalyst. The sodium hydroxide leaching selectively extracted more than 90% Mo or W, and less than 34% Al. The Al extraction was held to less than 0.1% for the Ni-Mo-Al catalyst, and 13% for the Ni-W-Al catalyst by the appropriate selection of the NaOH leach solution concentration and ratio of catalyst to leach solution. In both cases the Mo

TABLE III. - Metal extraction from various catalysts by leaching

Spent catalyst	Leaching reagent, g/L	Extraction, %									
		50 g catalyst/ 1 L reagent					100 g catalyst/ 1 L reagent				
		Ni	Mo	W	Cu	Al	Ni	Mo	W	Cu	Al
Ni-Mo-Al(B)	100 NH_4OH + 300 $(NH_4)_2SO_4$	74	90	NAp	NAp	0.2	90	89	NAp	NAp	0.1
Ni-W-Al	.....do.....	44	NAp	64	NAp	.04	(1)	(1)	(1)	(1)	(1)
Ni-Cu-Si	.....do.....	10	NAp	NAp	72	NAp	(1)	(1)	(1)	(1)	(1)
Ni-Mo-Al(B)	10 NaOH.....	.06	92	NAp	NAp	10	.02	79	NAp	NAp	.05
Ni-Mo-Al(B)	20 NaOH.....	.08	96	NAp	NAp	34	.08	92	NAp	NAp	.06
Ni-W-Al	10 NaOH.....	.02	NAp	86	NAp	15	(1)	(1)	(1)	(1)	(1)
Ni-W-Al	20 NaOH.....	.03	NAp	91	NAp	25	.02	NAp	87	NAp	13
Ni-Mo-Al(B)	50 H_2SO_4.....	94	88	NAp	NAp	68	86	81	NAp	NAp	33
Ni-Mo-Al(B)	100 H_2SO_4.....	98	93	NAp	NAp	96	93	88	NAp	NAp	61
Ni-W-Al	50 H_2SO_4.....	77	NAp	44	NAp	42	(1)	(1)	(1)	(1)	(1)
Ni-W-Al	100 H_2SO_4.....	94	NAp	28	NAp	76	71	NAp	41	NAp	41
Ni-Cu-Si	50 H_2SO_4.....	81	NAp	NAp	89	NAp	75	NAp	NAp	87	NAp
Ni-Cu-Si	100 H_2SO_4.....	91	NAp	NAp	90	NAp	94	NAp	NAp	89	NAp

NAp Not applicable. [1]Not looked for.

or W extraction still remained near 90%. Sulfuric acid leaching was effective for Ni-Mo-Al and Ni-Cu-Si catalysts but not as effective for the Ni-W-Al catalyst. With this approach, more than 90% of the Ni, Mo, and Cu, and less than 44% of the W were extracted. Over 90% of the Al was extracted with 100 g/L H_2SO_4 and 50 g catalyst/1 L reagent. If there were an incentive to recover Al, the sulfuric acid leaching approach is very effective for the Ni-Mo-Al catalyst, although a large amount of acid would be required. Sulfuric acid leaching was only effective in extracting Ni from Ni-W-Al catalyst, but the W can be extracted with a sodium hydroxide leach prior to the Ni extraction.

The sodium carbonate roasting approach was found to be very selective for extraction of Mo, W, or Cr as shown in Table IV. More than 90% of the Mo or Cr and 77% of the W were extracted. This approach was found to be the most promising approach for the Cr-Fe catalyst in early research. However, a sodium hydroxide roasting approach shows some promise for the Cr-Fe catalyst and is currently being evaluated. The sodium carbonate roasting approach for Ni-Mo-Al or Ni-W-Al catalysts could not compete with the sodium hydroxide leaching approach. The former roasting approach requires too much energy during the roasting process.

TABLE IV. - Metal extraction from various catalysts by sodium carbonate roasting followed by water leaching

Spent catalyst	Extraction, %					
	Ni	Mo	W	Al	Cr	Fe
Ni-Mo-Al.....	0.1	99	NAp	3	NAp	NAp
Ni-W-Al.....	NAp	NAp	77	2	NAp	NAp
Cr-Fe.....	NAp	NAp	NAp	NAp	92	0.004

NAp Not applicable.

Metal separation data obtained by solvent extraction with various extractants are shown in Table V. Extractants were identified for extraction of Ni^{++}, Cu^{++}, MoO_4^{--}, WO_4^{--}, and CrO_4^{--} in basic or acid solutions. LIX 64N extractant selectively extracted Ni^{++} in basic and Cu^{++} in acid

TABLE V. - Metal separation from leached spent catalyst pregnant solutions by solvent extraction.

Spent catalyst	Process	Extractant, vol %	pH	Extraction, %		
				Ni	Mo	Al
Ni-Mo-Al	Chlorination	5 Kelex 100 + 25 LIX 64N + + 70 Kerosene	3.6	14.8	ND	0.3
	NH_4OH $+(NH_4)_2SO_4$	5 Aliquat 336 + 2 Isodecanol + 93 Kerosene	10	.2	13.7	ND
		10 LIX 64N + 90 Kerosene	10	68.5	.3	ND
	H_2SO_4	5 Alamine 304 + 2 Isodecanol + 93 Kerosene	1.1	.3	99.7	.2
		15 DNNSA + 20 LIX 63 + + 5 Isodecanol + 60 Naphtha	1.1	68.5	70.2	.1
	NaOH	5 Aliquat 336 + 2 Isodecanol + 93 Kerosene	12.8	ND	46.0	3.6
				Ni	W	Al
Ni-W-Al.	Chlorination	5 Kelex 100 + 25 LIX 64N + 70 Kerosene	4.0	14.9	ND	0.5
	NaOH	5 Aliquat 336 + 2 Isodecanol + 93 Kerosene	13.4	ND	48.7	.1
				Ni	Cu	
Ni-Cu-Si	H_2SO_4	10 LIX 64N + 90 kerosene	2.5	0.06	88.7	
				CrO_4	CO_3	
Cr-Fe...	Na_2CO_3	5 Aliquat 336 + 2 Isodecanol + 93 Kerosene	12.6	57.5	4.9	

ND Not determined.

solution, and a combination of Kelex 100 and LIX 64N selectively extracted Ni^{++} in acid solution. Aliquat 336 selectively extracted MoO_4^{--}, WO_4^{--}, and CrO_4^{--} in basic solutions, and Alamine 304 selectively extracted MoO_4^{--} in acid solution. A combination of DNNSA and LIX 63 coextracted Ni^{++} and MoO_4^{--} or Ni^{++} and Cu^{++} in acid solution, and Ni^{++} and Al^{+++} in aqueous chloride solution. The TBP or naphthenic acid did not extract Ni^{++} or Cu^{++} in acid solution.

Based on the chlorination, leaching, and roasting approaches and metal separation tests, suitable processing schemes were proposed for Ni-Mo-Al, Ni-W-Al, Ni-Cu-Si, and Cr-Fe catalysts, as illustrated in Figures 2-5, respectively. Figure 2, for the Ni-Mo-Al catalyst, is a typical chlorination process scheme that can be applied to other hydroprocessing catalysts. The catalyst was roasted prior to the chlorination. The roasted catalyst was chlorinated with chlorine and air, and the chlorinated spent charge was hydrolyzed. The final products were recovered as follows: MoO_3 was recovered by dechlorination of the MoO_2Cl_2, Ni by solvent extraction followed by electrowinning, and Al_2O_3 by precipitation followed by calcination. Although Ni and Al were separated successfully by the preceding scheme, alternative approaches are being evaluated to improve separations. These schemes include separation of Al by precipitation as $Al(OH)_3$ and Ni by formation of $Ni(NH_3)_6^{++}$ with ammonium hydroxide-ammonium chloride.

A dual $NaOH-H_2SO_4$ leaching processing scheme, Figure 3, was applied to the Ni-W-Al catalyst and potentially can be applied to other hydroprocessing catalysts. Within this approach the catalyst was leached with sodium hydroxide, the WO_4^{--} ion in the filtrate was precipitated as $CaWO_4$ with $CaCl_2$, the leached residue was releached with sulfuric acid, Ni was recovered by solvent extraction followed by electrowinning, and Al_2O_3 was formed by precipitation followed by calcination.

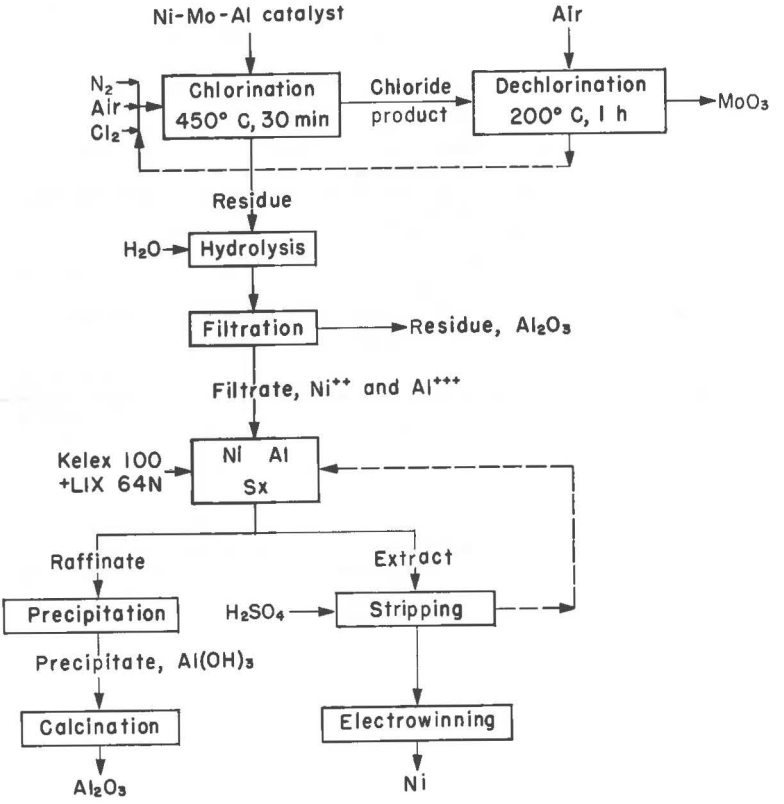

FIGURE 2 - Flowsheet for recovery of Ni and Mo from spent Ni-Mo-Al catalyst by chlorination.

The sulfuric acid leaching approach, shown in Figure 4, was found to be the most promising approach for recovering Ni and Cu from the Ni-Cu-Si catalyst. In this approach the catalyst was leached with sulfuric acid. Subsequently the leached solution was filtered and the Ni^{++} and Cu^{++} in the filtrate were separated by solvent extraction. The final Ni and Cu products were recovered by electrowinning.

The sodium carbonate roasting process scheme for the Cr-Fe catalyst is shown in Figure 5. The catalyst was roasted with sodium carbonate and then leached in water. The CrO_4^{--} in the filtrate was extracted by solvent extraction. The final Na_2CrO_4 product was recovered by evaporation and crystallization. An alternative approach is being evaluated for recovering Cr from the Cr-Fe catalyst by first reducing CrO_4^{--} to Cr^{+++} with Na_2SO_3 or N_2H_4 and then precipitating the Cr^{+++} with NaOH.

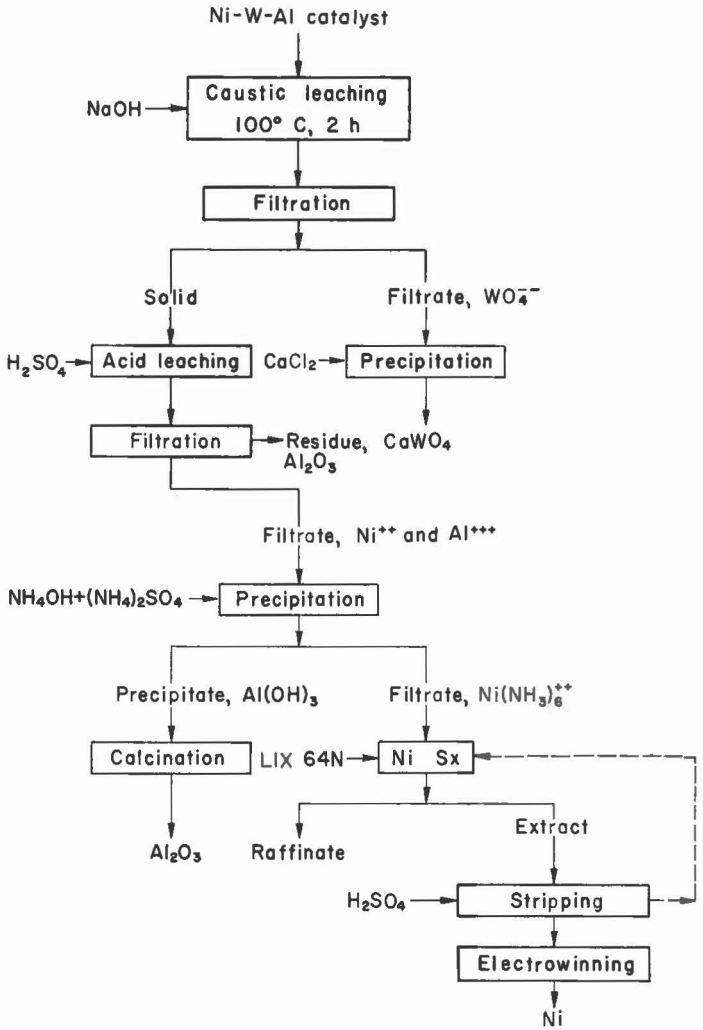

FIGURE 3 - Flowsheet for recovery of Ni and W from spent Ni-W-Al catalyst by sodium hydroxide-sulfuric acid leaching.

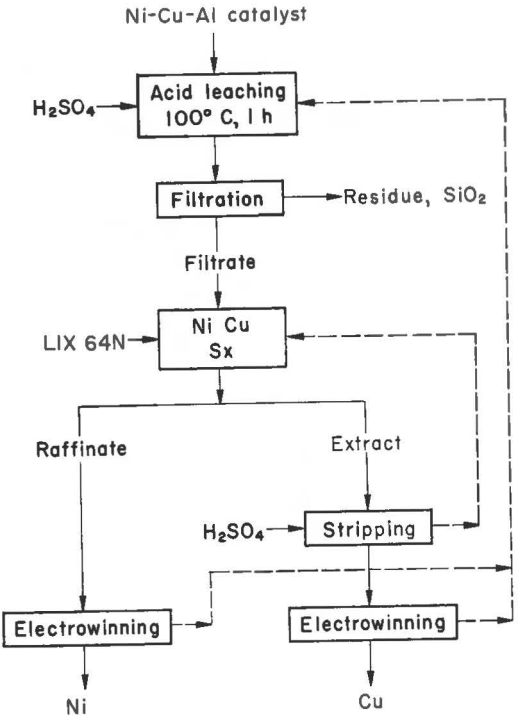

FIGURE 4 - Flowsheet for recovery of Ni and Cu from spent Ni-Cu-Si catalyst by sulfuric acid leaching.

The economic feasibiity of the proposed process schemes is being evaluated by the Bureau's Process Evaluation Group. The recovery schemes and impurity levels in the final products are being evaluated at the Albany Research Center. Final results of the research will be published in a future Bureau of Mines Report of Investigations.

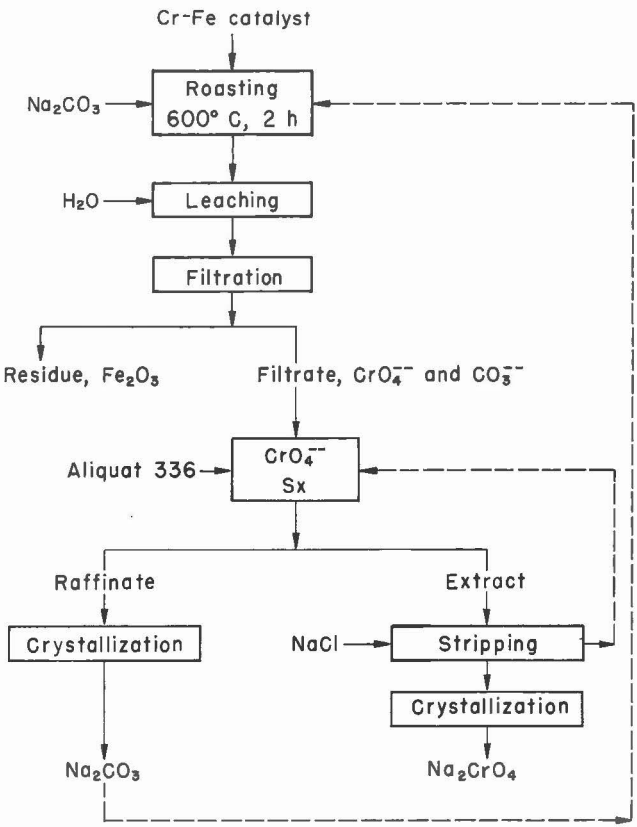

FIGURE 5 - Flowsheet for recovery of Cr from spent Cr-Fe catalyst by sodium carbonate roasting followed by water leaching.

Summary and Conclusions

Studies indicated that 73 to 99% of the metals in certain spent catalysts can be extracted by a variety of approaches. Organic extractants that will separate metal ions in solution were identified, and total processing schemes for recovering metals from spent Ni-Mo-Al, Ni-W-Al, Ni-Cu-Si, and Cr-Fe catalysts were proposed. Final recovery schemes need to be optimized, and impurity levels in the final products need to be established. The economic feasibility of the proposed processing schemes is being evaluated.

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