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The Recovery of Metal Ions from Spent Catalysts: A Study of Desulfurization Catalysts

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Abstract – The catalysts used in refining processes are regularly deactivated over time. Typically, the operation of the catalyst can be regenerated and reused, although it is not possible to recycle the catalyst always. Following a few regeneration and reuse cycles, the operation of the catalyst will decrease to very low levels and further regeneration mechanism cannot be achieved economically. In solid waste type, the expended catalysts are disposed of. The disposal of spent catalysts includes respect of rigorous legislation on the climate. Because of the self-heating activity and toxic chemical content, spent hydrotreating catalysts is listed by the Environmental Protection Agency as hazardous waste in the USA. Study on the advancement of processes for the processing and reuse of waste hydrotreating catalysts has resulted in strict environmental controls on the storage of and disposal of spent catalysts. The current study is about recuperation of metals, in particular molybdenum and cobalt from spent catalogs used in desulfurization by leaching using solutions of caustic soda and sulfuric acid. The results have been studied for the purpose of fixing apological solution with the five experimental parameters (leaching solution concentration, solid to liquid ratio, particle size, turbidity and temp). The suggested requirements are 1M for the NaOH solution and 1/100 for H2SO4 solution, 1/100 ratio of strong catalyst mass in grams to leaching solution volume in milliliters, rhythmic rhythm of agitation of 250 rpm, particulate size less than 150 µm and temperature less than 80°C. The conditions are 47,092% molybdenum extraction and 72, 73% cobalt extraction.

Keywords – Metal Ion, Spent Catalyst, Desulfurization

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INTRODUCTION

Catalysis is extremely important for chemical processes. An approximate 90% of the processes involved in chemical processing are focused on catalytic technology covering four main industry segments: fuel refining, polymerizations, production of chemicals and environmental remediation. The catalytic devices are disabled over time, so after a few cycles of restoration and reuse, the operation of the catalyst may be decreasing to very low levels. Further regeneration may not be commercially viable. Where there is a deterioration of the activity of the catalytic converter, it is normally regenerated and reuse. The spent catalysts are recycled as solid waste and are rising every day their exposure to environmental pollution. In a number of nations, the dangerous existence of the expended catalytic systems attracts concern by the environmental authorities. The United States Environmental Protection Agency has listed spent catalysts as toxic waste (USEPA). At the same time, the majority of these catalysts include platinum grouped metals (PGMs), including: nickel (Ni), vanadium (V), molybdenum (Mo) and cobalt (Co), which is supported in oxides or sulphide such as Alumina and silica in the form of: platinum (Pt), palladium (Pd), rhodium (Rh, Iridium (Ir). Recovery of the metals used to make ferro-alloys and other useful materials in the manufacture of carbon and stainless steel as an alloying agent is an attractive solution.

The focus is on extracting these metals from catalytic converters for automobiles, petroleum catalysts, petrochemical and Pharmaceutical industries, in the area of metal recovery described above. Recovery techniques are categorized in different forms such as organic solvent extraction, ion exchange resins, carbon adsorption and membrane use. Many of these systems have a huge number of chemical reactions, including more than one processing unit and advanced machinery for removing these metals, which are quite expensive. These techniques are therefore categorized by three categories in several of these sources: pyrometallurgy, hydrometallurgy and electrometallurgy. Metal to gas conversion and bioleaching approaches are often possible as retrieval techniques for metals in addition to the above methods. Depending on the number and the kind of metals in the catalyst, their concentration, catalyst base nature and overall catalyst quantity, the choice of method depends. The electrolysis method includes the range and basic tools, quick to work, cost-effective and high recovery with highly pure metals, among the techniques used to separate metals.

LITERATURE REVIEW

Venkateswaran, et. al. (2010) The nickel electrolyte was tested. In order to improve nickel deposition, they investigated the effect of electrolyte pH, current density, temperature, and supplement agents (boric acid). Lower chloride energy intake in comparison to sulfate largely reflects improved electrolyte conductivity and increased electrode reversibility.

Lupi, C. and Pasquali, M. (2013) The electrolysis and electrolysis method for LiCoxNi1-xO2 Ni-metal and powder recovery has been investigated, respectively with constant current density and constant potential. Nickel electrolyte provides a strong feature of current performance Ni deposit and an electronic intake of about 87 percent and 2,96 kWh/kg at 250 A/m2, ph=3–3.2 and an electrical composition of about 50 g/l Ni and 20 g/l H3BO3. With a continuous solution of 1,7–1,8 g/l NI, electrolysis creates extremely pure powder at 80 minutes, which leaves less than 100 ppm in solution.

Kirshin, M.Yu. and Pokhitonov, Yu.A. (2015) Studied palladium deposition from a platinum electrode nitrate solution. The Pd deposition intensity of 1 M HNO3 solution was 0.7-0.9 mg/cm-2 with a current efficiency of around 70 percent inside the E theoretical range of +0.5 to +0.25 V. The Pd recovery level is greater than 99 percent by cathode deposition.

Garcia-Gabaldon, M. et. al. (2015) The impact of Pd (II) on electrochemical deposition of Sn(IV) was evaluated. They analyzed the conversion fraction, current performance, time output, and the electronics reactor's basic energy usage. At -0.6V, comparatively low overall energy consumed values were achieved, with a global current performance of 74%. In the other side, real strong values for palladium (72%) and tin (73%) have been achieved for this opportunity and t100 fractional conversion.

Dorikar, D.K. et. al. (2016) Nickel sulphate bath electrodeposition in ammonia medium has been studied. With increasing acetone concentrations up to 15 percent V/V in bath fluid, existing performance has been found to be increasing. The stability of the deposited electro-nickel powder is between 85 and 89%. As the level of acetone rises, the average particle size of the deposited powder was declining from 13 to 16 μm.

Lai, Y-C et. al. (2018) The acid leaching and fluidized-bed electrolysis method was studied with useful metal recovery from expensive hydrodesulfurization (HDS). Acid B was determined to be higher than acid A in acid leaching metals (HNO3/H2SO4 = 1:1) with a volume ratio of

HNO3/H2SO4/HCl = 2:1:1. At 70oC, for acid B, respectively, the best S/L ratio and duration of washing are 40g/L and 1 h. For the goal metals Mo, Ni and V, respectively, the first leach rates hit 90, 99 and 99%. The electrolyte recovery of Mo, Ni and V at 2 h was 15, 61, and 66 percent, respectively, with the use of acids B at S/L = 40 g/L, one phase leaching at constant current density=35.7 mA/cm2 with a stable cell voltage of 5 V.

Jayakumar, M. et. al. (2009) Studies in platinum acid and in stainless steel electrodes in cyclic voltammetry acid medium of palladium electrolysis (II) is performed. In the case of electrolysis from simulated high-level liquid waste, recovery and Faradaic performance were dramatically decreased (to 40%) due to more competitive reactions.

Nathan, W. and Michael, L.F. (2009) The rest of the palladium was examined by electrochemical deposition on a cathode high surface area, from spent electroless bond pad plating solution. They proposed their process of recycling as an economic and ecological mechanism.

Prajapati, R.P. et. al. (2010) Used nickel recovery electro-deposition from the spent nitrogen fertilizer catalyst sector. Their findings showed that the volume of nickel recovered increases with the initial nickel level, temperature and deposition period.

MATERIALS AND METHODS

Sample Preparation

The waste-solid material taken arbitrarily from ANRPC refinery stores was the sample of the spent catalyst (Como/Al2O 3) used in these tests (3 kg). The catalyst type was cylindrical and contained 4 mm bis 2 mm of particle size. A deionized water cleaned and dried at 110 °C. The sample was removable. This sample was then roasted in a 650°C oxygen gas.

Leaching Study

A conical 1000 ml flask was used as a reaction reservoir, mounted over a water bath with a shaker supplied by operated electrical heater, to perform leaching experiments. Disposing and diluting (1-4 percent) of solid NaOH was used to provide the appropriate (1-4) molar solution of caustic soda for 0.5 liters and Dilution of (98 wt. percent) H2SO4 was used to develop the necessary (0.25-1) molar solution of sulfuric acid per 0.5 litter. In the temperature controller decimal, the operating temperature of the laboratory (30- 80 °C) was set. Until it is introduced into the reactor to reduce the instable heating period, the solution of caustic soda and sulfuric acid was chopped down to the required temperature (30- 80 °C). Shaker was placed at the requested rpm mixing velocity (50-250). The 0,5 L of caustic soda or sulfuric acid solution has been applied and added to the shaking unit 5 g of expended catalytic at the desired mesh size. Turn on the shaker and see the stop. The sample of 5 mL solution was removed and purified after the desired duration (5-120 minutes). Samples is tested for solution from both tests using Atomic Spectroscopy for molybdenum and cobalt. Any of the lixiviation experiments in current research have been replicated for reproductivity and an experimental loss of ±2%.

Analytical Methods

A powder diffractometer was used for X-ray examination of the initial spent catalyst sample. Fig.1 displays the variations of X-ray diffraction from the initial study of the spent catalyst (Burker-xrd D2 PHASER). The figure shows that the spent catalyst comprises essentially metal ions Mo and Co in their oxide shape.

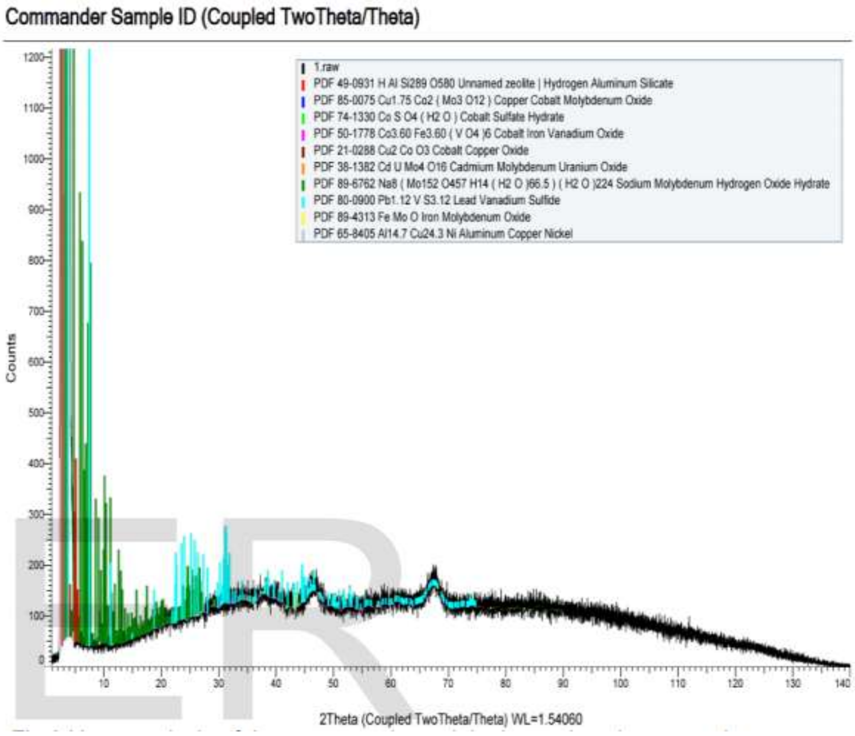
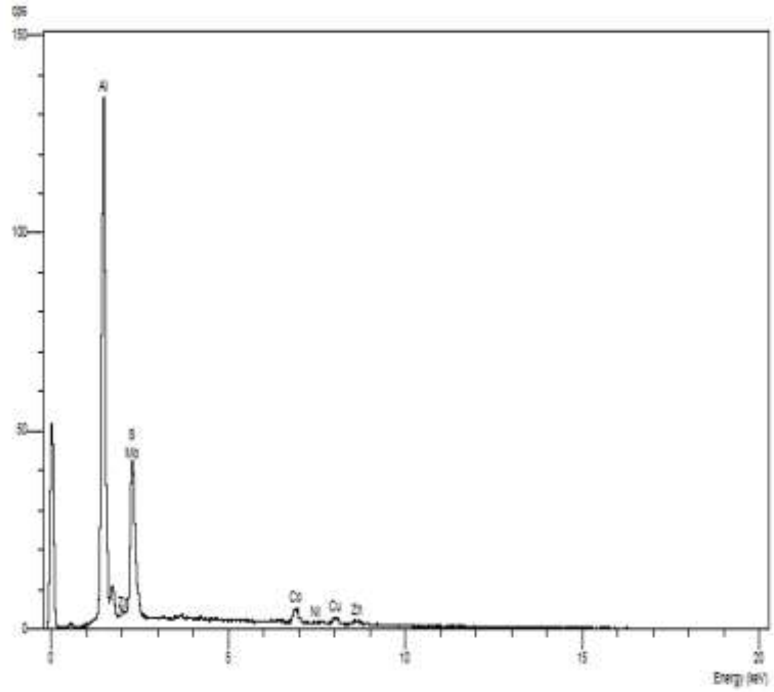


Figure 1: Analysis of the initial spent catalyst with a powder diffractometer

In order to analyze the mineralogic species in the spent catalyst, the electron scanning microscopies (SEMEDX) with an energy dispersive spectrometer (JEOL JSM-5300) were conducted. As seen in fig.2, the integrated Sulphur form was observed in the molybdenum.



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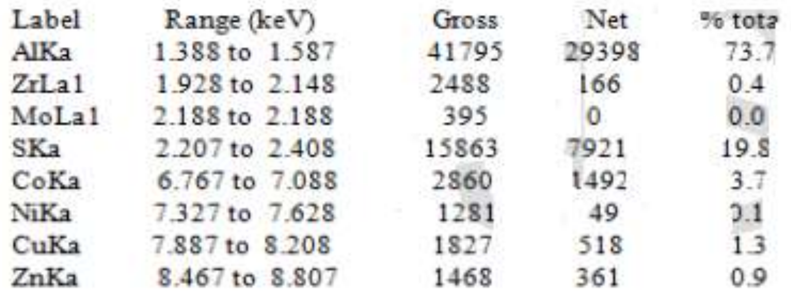
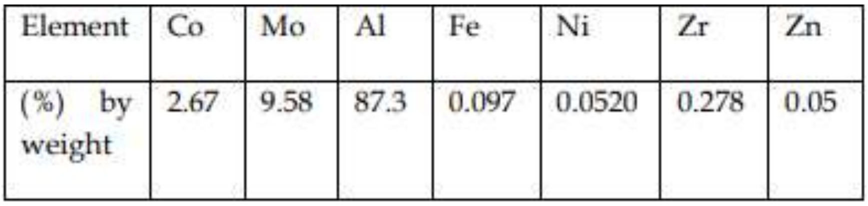


Label	Range (keV)	Gross	Net	% tota
AlKa	1.388 to 1.587	41795	29398	73.7
ZrLa1	1.928 to 2.148	2488	166	0.4
MoLa1	2.188 to 2.188	395	0	0.0
SKa	2.207 to 2.408	15863	7921	19.8
CoKa	6.767 to 7.088	2860	1492	3.7
NiKa	7.327 to 7.628	1281	49	0.1
CuKa	7.887 to 8.208	1827	518	1.3
ZnKa	8.467 to 8.807	1468	361	0.9

Figure 2: The analysis of the original spent catalyst sample

Xray fluorescence (Rigaku NEX CGEDXRF) for the examination of the elemental compositions of the original expensive catalyst. The scan was performed using an electron microscopy with an energy dispersive spectrometer (SEMEDX). The basic compositions are mentioned the initial spent catalyst

Table 1. Chemical Compositions of Spent Catalyst



Element	Co	Mo	Al	Fe	Ni	Zr	Zn
(%) by weight	2.67	9.58	87.3	0.097	0.0520	0.278	0.05

The spectroscopy of atomic absorption examined all aqueous samples generated during the leaching analysis of the spent catalyst. Digestion by using condensed HF accompanied by dilution and examination of the initial spent catalyst was completed. Measured by the atomic absorption spectroscope the concentration of metal ions Mo and Co in the above described solution (ContrAA 300).

RESULTS AND DISCUSSION

Leaching of Spent Catalyst with NaOH

The effect of NaOH on Mo recovery

Four concentration levels have been chosen (1, 2, 3, 4 M). Expertise has been conducted at constant conditions, when the temperature is 30 °C (S/L mass) and when the solid mass is 1/100 (g/mL), the rhythm of rise is 150 rpm (g/ml) and the particle size is below 150 μm to research the percentage of molybdenum recovery from the NaOh solution concentration effect. The data showed in Fig.3 shows that raising the NaOH solution concentration above 1 molar is not significantly beneficial to the molybdenum recovery, as the NaOH solution recovers at 2 molar caustic soda in approximately 1 percent, when concentrations over 2 M are slightly lower, which can be explained by the occurrence of different levels of NaOH. Therefore, 1 molar NaOH solution is regarded as the strongest solution in which around 26,976% of molybdenum recovered.

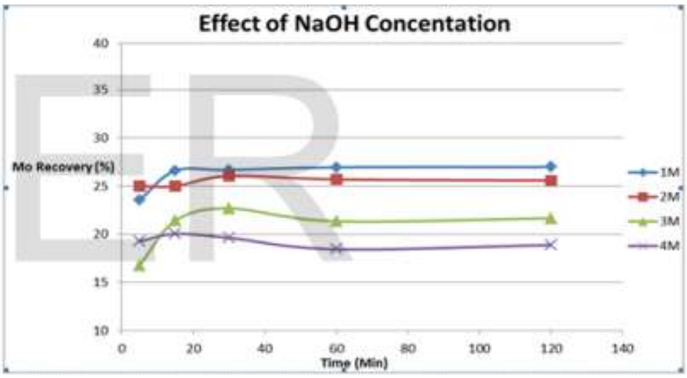


Figure 3: Recovery for Mo effect of NaOH concentration Ring speed=150 rpm, particle size= <150µm (Temperature= 30°C, S/L= 1/100(g/ml).

The effect on Mo recovery of the solid to liquid ratio (S/L). Five concentrations were calibrated for the solid/liquid ratio ((1/10) -(1/100)) of solids in grams and fluid in ml/ml. The experiments were performed under constant conditions, in which the temperature of the solid to liquid relation on the percentage of the molybdenum recovered was 30°C, with a concentration of 1 M NaOH, a speed of 150 rpm, and with a particle size of <150µm. In figure 6, data reveal that the S/L ratio decline results in a significant growth in the percentage of recovered molybdenum. Therefore, the best ratio of 1 gram of the expended catalyst/100 ml is considered whereby 26,976 percent of molybdenum recovers.

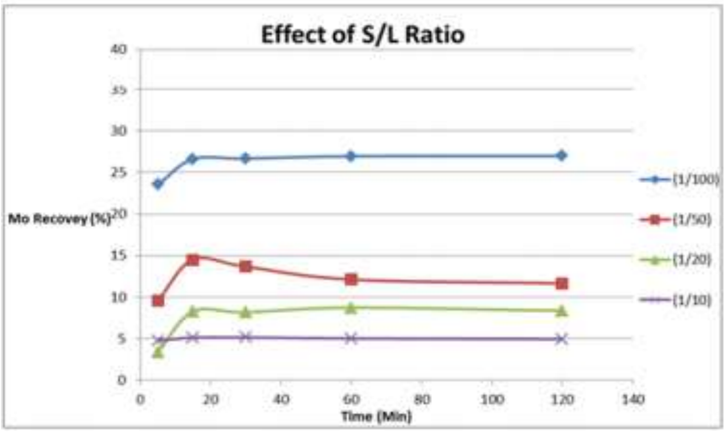


Figure 4: Effect of RPM (temperature=30°C, NaOH concentration=1M, RM=150 rpm, scale of the particle=<150µm)

The Effect of Mo Recovery Stirring

Three agitated speed ranges were analyzed in the range (50 to 250 rpm). The tests were undertaken at constant temperatures, under which the temperature was 30°C, the concentration of NaoH was 1M and the solid to liquid ratio (1/100) was solid mass grams, and the liquid volume in milliliters and the particle size in the molybdenum recovery was under 150µm. The figure 5 reveals that the recovery from molybdenum increases by increasing the rush. Therefore, the highest performance for molybdenum retrieved is at a pace of 250 rpm, about 32.55%.

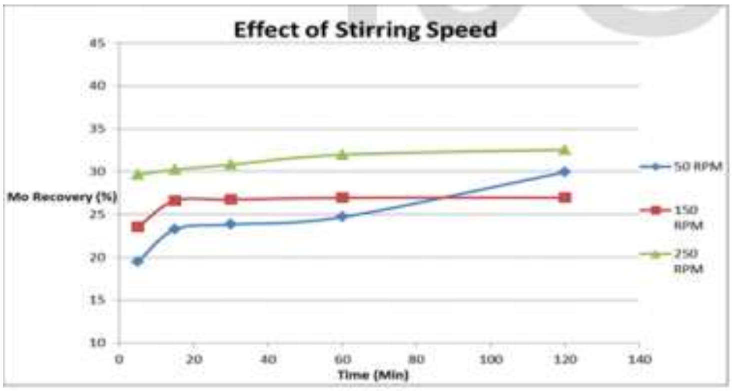


Figure 5: Remove velocity effect on Mo recovery "S/L= 1/100 g/ml, particulate size=<150µm) (Temperature= 30°C, NaOH Concentration = 1M)."

Temperature effect on Mo recovery

The studies have been conducted with three separate temperature ranges (30 -80 °C). The tests were performed under constant conditions with a mixing speed of 150 rpm, a NaOH level = 1M, a solid-to-liquid ratio (1/100) of solid mass, and a liquid volume of milliliter (g/ml) and a molybdenum regeneration particle size <f. The figure6 indicates that the recovery of molybdenum by rising temperature is notablely increasing. Thus 80°C is deemed the best temperature at which 47,092 percent of the molybdenum is retrieved.

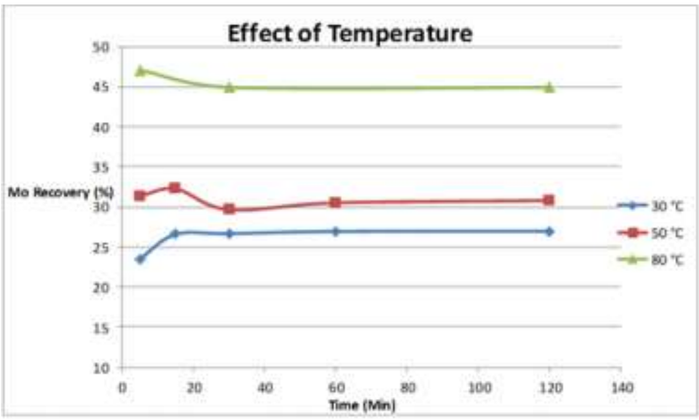


Figure 6: Temperature impact on Mo recuperation ("Stirring speed= 150 rpm, NaOH Concentration= 1M, S/L= 1/100 (g/ml), Particle size=< 150µm")

Spent Catalyst Leaching with H2SO4

The effect of NaOH on Co recovery

4 standards have been chosen (0.25, 0.5, 0.75, 1 M). Solid mass (1/100) is solid and liquid volume in milliliters (g/ml), stirring speed is 150 rpm and particle size <150µm for the analysis of the solvent sulfuric acid (H2SO4) concentrations impact on the percentage of cobalt recovery were carried out under constant conditions. The figures in fig.6 demonstrate that rising sulfuric acid concentrations above 0.5 molar may not have a major beneficial impact on cobalt recovery, since cobalt recycling is equal to or marginally higher since it is more likely to collision with high H2SO4 concentrations. The results are seen in fig.7. Therefore, 0.5 molar sulfuric acid is regarded as the maximum at which around 55,304 per cent of cobalt reclaimed.

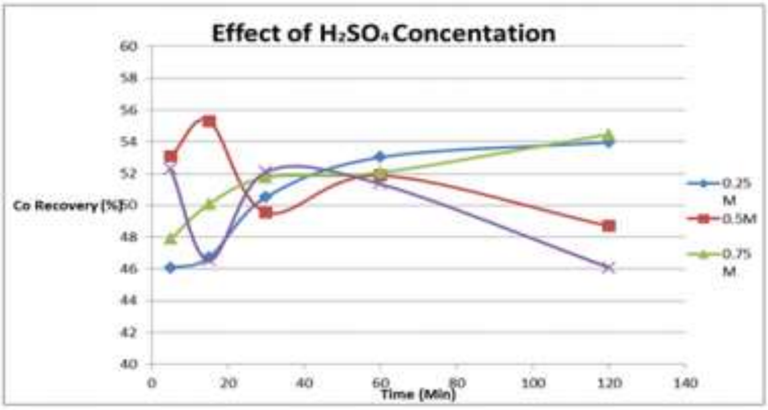


Figure 7: Recovery effect of H2SO4 concentration (“Temperature= 30°C, S/L= 1/100(g/ml), Stirring speed=150 rpm, Particle size=< 150µm”)

The impact on co recovery of the particle size

From particles of over or equivalent to 850 µm to particles of less than 150 µm were chosen four ranges of particular scale. Solid to liquid ratio (1/100) is (1/100) where solid mass is grams and the fluid mass is in millilitres (g/ml) and the velocity at which stirrings were carried out is 150 rpm, to research the influence of the particle size on the cobalt recuperation percentage. The figure8 reveals that the smaller the particle volume, the higher it recovers. The particle size < 150 µm is therefore deemed the most optimal, when around 55.304 percent of the recovered cobalt accounts.

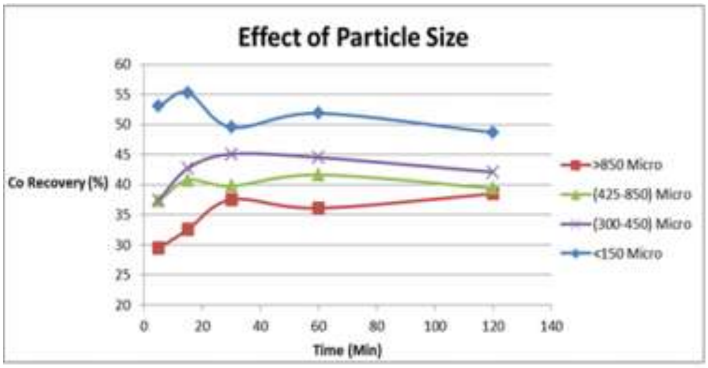


Figure 8: co Recovery effect of particle size (“Temperature= 30°C, H2SO4 Concentration= 0.5 M, Stirring speed=150 RPM, S/L= 1/100(g/ml”))

Solid to Liquid Ratio (S/L) effect on co recovery

A strong to liquid relationship is a key factor to be analyzed such that the best ratio can be established in order to achieve optimal cobalt recovery and ensure adequate supply without waste in raw materials. Five concentrations were calibrated for the solid/liquid ratio ((1/10)-(1/100)) of solids in grams and fluid in ml/ml. The tests have taken place in constant conditions at 30°C, with the H2SO4 content 0.5M, 150 rpm and a particle size <150µm to research the influence of a solid to a liquid relationship on the percentage of cobalt recovery. The experiments have also taken place under constant conditions. The figure 9 indicates a strong rise in percent of recovered molybdenum as a result of the reduction in the S/L ratio. The 1 g/100 mL ratio of sulphurice solution, where cobalt recovered accounts for about 55,304 percent, is considered the optimal ratio of 1 g/100 ml.

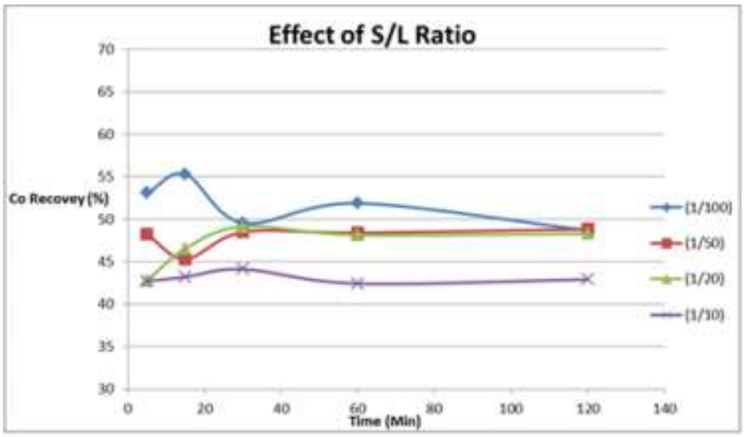


Figure 9: Effect of S/L ratio on Co recovery (“Temperature= 30°C, H2SO4 Concentration= 0.5 M, Stirring speed=150 rpm, Particle size= <150µm”)

Stirring speed's impact on Co recovery

Three agitated speed ranges were analyzed in the range (50 to 250 rpm). Solid to liquid ratio of 0.5M (1/100) while solid mass is in grams, and liquid volume is in milliliters (g/ml), and the particle size <150 µm to analyze stirring speed impact on the molybdenum recovery rate, were tested under consistent conditions where temperature is 30 °C, H2SO4 concentration is 0.5 M. The details in fig.10 indicate that the recovery of cobalt is rising by the rate of agitation. Stirring speed of 250 rpm is thus deemed the optimal for the restoration rate of 61.304% of cobalt accounts.

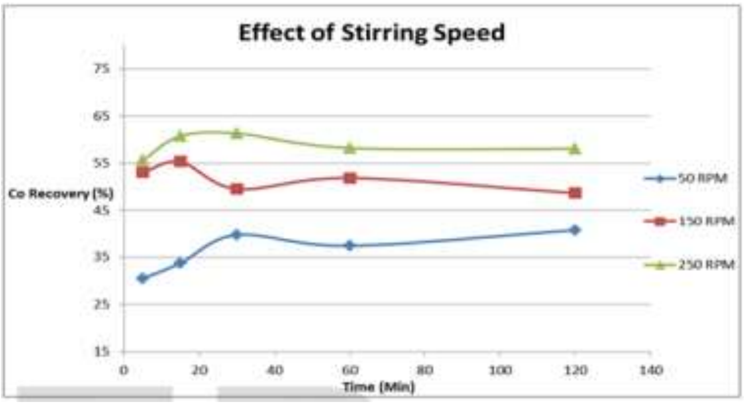


Figure 10: Stirring pace effect on Co recovery (“Temperature= 30°C, H2SO4 Concentration= 0.5 M, S/L= 1/100 (g/ml), Particle size= <150µm”)

Temperature impact on recovery

The tests selected four distinct temperature ranges (30 – 80 °C). Screening speed 150 rpm, concentration of H2SO4 0.5M (1/100) and solid to liquid ratio (1/100) is performed during experiments where solid mass is in gram, liquid volume is in milliliter (g/ml) and the particle size is <150µm to analyze temperature impact on cobalt recovery percentages. The figure 11 evidence indicates that the cobalt recovery by rising temperature has significantly increased. Therefore, a maximum temperature of 80°C as cobalt recovered accounts for approximately 72,73%.

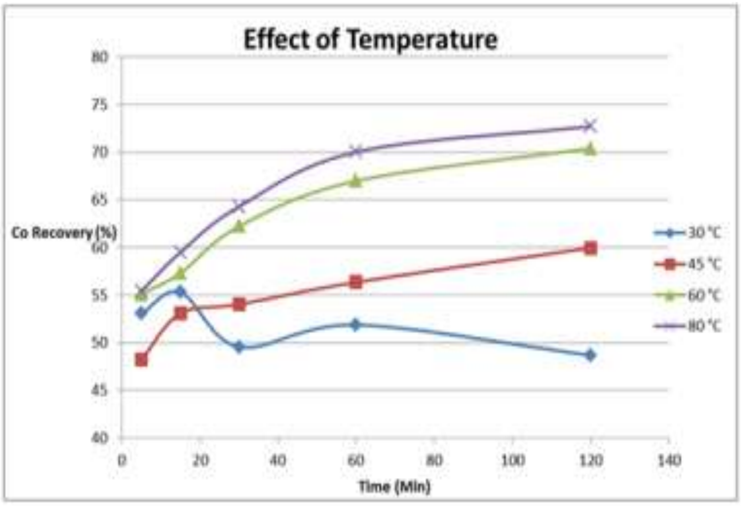


Figure 11: Temperature impact on Co recovery (“Stirring speed= 150 rpm, H2SO4 Concentration= 0.5 M, S/L= 1/100 (g/ml), Particle size=<150µm”)

The impact of NaOH consents, catalyst in grams to the liquid volume of NaOH in milliliters (g/ml) (S/L) solution, Stirring speed and temperature on the kinetics of the response is shown in the table 2.

TABLE 2 Molybdenum Leaching Rate Determination Constant

Component	Constant conditions	Main parameter	Value	Rate constant (K) (ppm/min)
Molybdenum (Mo)	Temp = 30°C S/L = 1/100(g/mL) Stirring Speed=150RPM Particle Size < 15Micro	NaOH Concentration	1M	110.89
			2M	105.86
			3M	89.17
			4M	78.23
	Temp = 30°C Conc = 1M Stirring Speed=150RPM Particle Size < 15Micro	Solid / Liquid ratio	(1/10) g/ml	20.74
			(1/20) g/ml	34.41
			(1/50) g/ml	50.34
			(1/100) g/ml	110.89
	Temp = 30°C Conc = 1M S/L = 1/100(g/mL) Particle Size < 15Micro	Stirring speed	50 rpm	112.04
			150rpm	110.892
			250rpm	132.06
	Conc = 1M S/L = 1/100(g/mL) Stirring Speed=150RPM Particle Size < 15Micro	Temperature	30 °C	110.89
			50 °C	126.81
			80 °C	157

Table 3 displays the effect of the H2SO4 concentration, spent catalytic particle size, catalytic mass in grams to the liquid volume of H2SO4 solution in milliliters (S/L) in milliliters (g/ml). Stirring speed and kinetical temperature to calculate the rate at which the euphuizing catalyst produces cobalt.

TABLE 3 Determining cobalt leaching response rate constants

Component	Constant conditions	Main parameter	Value	Rate constant (K) (ppm/min)
Cobalt (Co)	Temp = 30°C S/L = 1/100(g/mL) Stirring Speed=150RPM Particle Size < 15Micro	H2SO4 Concentration	0.25M	5034
			0.5M	4798.5
			0.75M	5075.7
			1M	4630.3
	Temp = 30°C Conc = 0.5M Stirring Speed=150RPM S/L = 1/100(g/mL)	Particle size	>850µm	3554.4
			(425-850) µm	3835.2
			(300-425) µm	4119.6
			<150 µm	5034
	Temp = 30°C Conc = 0.5M Stirring Speed=150RPM Particle Size < 15Micro	Solid / Liquid ratio	(1/10) g/ml	4108.8
			(1/20) g/ml	4604.1
			(1/50) g/ml	4631
			(1/100) g/ml	4798.5
	Temp = 30°C Conc = 0.5M S/L = 1/100(g/mL) Particle Size < 15Micro	Stirring speed	50 rpm	3729.4
			150rpm	4798.5
			250rpm	5608.8
	Conc = 0.5M S/L = 1/100(g/mL) Stirring Speed=150RPM Particle Size < 15Micro	Temperature	30 °C	4798.5
			45 °C	5505
			60 °C	6434.9
			80 °C	6675.2

CONCLUSION

The present research is about recuperating metals, in particular molybdenum and cobalt, from spent catalyst used in desulphurization phase by leaching using caustic soda and sulfuric acid solutions, in which the impact of five experimental parameters has been investigated (concentration of a leaching solution, solid/liquid ratio, particle size, agitating speed and temperature). The results of this analysis are as follows:

- Increased caustic soda concentrations above 1 molar or increased sulfuric acid concentrations above 0,5 molar have no major positive impact on molybdenum/cobalt recovery percentages.

- The reduction in the ratio of strong catalyst mass to the leaching solution's volume results in an improvement in the recovered percentage of molybdenum and cobalt. The ratio 1 gram expended catalyst is thus known to be prescribed with 100 ml of NaOH or H2SO4 solution.

- Molybdenum and cobalt recovery is increased by rising the rate of revitalization. Stirring speeds above 250 rpm are also deemed advisable.

- The smaller the scale of the particle, the more the molybdenum and cobalt are recuperated. The particle size < 150 μm is thus deemed advisable.

- The recovery of molybdenum and cobalt by rising temperature is clearly increasing. The temperature of 80°C is also deemed advisable.

- Conditions of optimal molybdenum-cobalt recovery in this analysis function with 1M for the NaOH solution and 0.5M for the solution H2SO4, (1/100) ratio of strong catalyst mass to the volume of the mililiter solution, 250 rpm stirring, 150 μm smaller and 80°C temperature. The conditions are 47,092% molybdenum extraction and 72,73% cobalt extraction.

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