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Extraction of Valuable Metals From Spent Hydrodesulfurization Catalyst By Two Stage Leaching Method

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Abstract

Spent hydrodesulfurization (Co-Mo/ γ -Al₂O₃) catalyst generally contains valuable metals like molybdenum (Mo), cobalt (Co), aluminium (Al) on a supporting material, such as γ -Al₂O₃. In the present study, a two stages alkali/acid leaching process was conducted to study leaching of cobalt, molybdenum and aluminium from Co-Mo/ γ -Al₂O₃ catalyst. The acid leaching of spent catalyst, previously treated by alkali solution to remove molybdenum, yielded a solution rich in cobalt and aluminium.

Keywords: spent HDS catalyst; sodium carbonate; sulfuric acid; leaching; valuable metals

Introduction

The petroleum refining industry makes extensive use of catalysts for desulphurization of various fractions. The most common hydrodesulphurization (HDS) catalysts are the Ni,Mo and the Co,Mo both on the γ -alumina support [1]. During hydrodesulphurization reactions, the catalysts are deactivated by compounds of S, C, V, Fe, Ni, Si and traces of As and P [2-4]. As a result, the spent catalysts are classified as hazardous materials [5]. Due to their toxic nature, the disposal of spent catalysts can pollute the environment since heavy metals are leached out. To avoid pollution in land disposal as well as to minimize landfill space, the spent catalysts are subjected to metal extraction by various solubilization processes and reused in a variety of applications [6-8]. However, such waste materials containing high metal

concentrations may be considered as "artificial ores" since they can serve as secondary raw materials with a consequent reduction in the demand for primary mineral resources. Recycling of spent catalysts became an unavoidable task not only for lowering catalysts costs but also for reducing their waste to prevent the environmental pollution [1]. Spent HDS catalysts generally consist of 10-30% molybdenum,1–12% vanadium, 0.5-6% nickel.1-6% cobalt. 8-12% sulphur, 10–12% carbon and the balance is alumina, which makes it economically viable for recovery of valuable metals [9]. During the past decade, more research work was conducted to recover molybdenum, vanadium and other valuable metals from spent HDS catalysts [10]. A number of processes and technologies for the recycling of valuable metals from spent catalysts have been

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developed and reported [11-15]. Basically, there are two main processes. *hydrometallurgical* and hydropyrometallurgical processes to recover valuable metals from spent *Hydrometallurgical* [16]. catalysts processes involves leaching with alkaline or acidic solutions. Many reagents, such as NaOH, H₂SO₄, NH₃, $(NH_4)_2SO_4$ and oxalic acid with H_2O_2 and $Fe(NO_3)_2$ have been tested [17,8] *Hydropyrometallurgical* processes such as calcination to remove the hydrocarbons, C and S followed by leaching with suitable reagent [18,19] or roasting of spent catalyst with Na₂CO₃, NaCl and NaOH at high temperatures followed by leaching [20,21,15] have been studied extensively for recovery of metals from spent HDS catalyst. The objective of the work presented in this study is intended to investigate the best operating conditions of metal removal (i.e. Mo, Co and Al) from spent Co- Mo/γ -Al₂O₃ catalyst by using two stages of extraction which contain alkali stage (i.e. sodium carbonate and hydrogen peroxide solution) and acid stage (i.e. sulfuric acid and hydrogen peroxide solution) [22].

Experimental

Materials

The following chemical agents were used in the experiments:

1. Spent Co-Mo/ γ -Al₂O₃ catalyst.

The spent Co-Mo/ γ -Al₂O₃ catalyst supplied from Al-Daura refinery was used. The shape of the catalyst was cylindrical (2.5mm x 1.5mm). Catalyst contained residue of oil and washed by acetone then by distilled water and dried overnight in an oven maintained at ~85°C. Finally, it was ground and sieved. For this study, two different size fractions of spent catalyst were prepared: (less than 75µm and greater than 75µm) and mixed thoroughly to obtain uniform sample for chemical analysis and leaching experiments. Chemical analysis of the examined samples are shown in Table (1). The analysis was done in Ibn Sina company by using atomic absorption analysis.

Table 1,	Chemical	analysis	of selected		
samples					

Cotolyst	Composition			
Cataryst	%Co	%Mo	%Al	
Co-Mo/ γ -Al ₂ O ₃	2.2	10.12	41.16	

- 2. Hydrogen peroxide (H₂O₂) supplied by Applichem GmbH Ollowg with assay of 50%.
- 3. Sulfuric acid (H_2SO_4) supplied by Sd fine-chem limited with a concentration of 98%.
- 4. Sodium carbonate (Na₂CO₃) Analar.

Apparatus

The apparatus used in the experiments was:

- 1. Water bath equipped with temperature controller (Baird and Tatlaock Unitemp water bath).
- 2. Mechanical mixer, which was calibrated using a digital tachometer (Victor DM6234P+).
- 3. Three neck round bottom flask (500ml) in volume.

The general arrangement of equipments used for extraction are shown in Fig. (1).



Fig. 1, Schematic diagram of the apparatus unit

Experimental design

Two stage leaching process was studied:

I- Study of Alkali leaching stage

In this study, sodium carbonate and hydrogen peroxide as extractant agents were used to extract Mo from spent catalyst. Different parameters for this leaching stage were studied as listed below.

- 1. Agitation time (30, 60, 90 and 120 minutes).
- 2. Agitation speed (100, 300 and 500 rpm).
- 3. Extractant concentration (sodium carbonate concentration of 40, 60, 80 and 120 g/l and hydrogen peroxide concentration of 5, 7, 10 and 12 vol.%).
- 4. Pulp density (5, 10, 20 and 30 w/v%).
- 5. Operating temperature (30, 50, 70 and 90°C)
- 6. Particle size (greater than 75 μm and less than 75 μm).

II- Study of Acid Leaching Stage

In this study, sulfuric acid and hydrogen peroxide as extractant agents were used to extract Co and Al. Different parameters for this leaching stage were studied.

- 1. Agitation time (30, 60, 90 and 120 minutes).
- 2. Agitation speed (100, 300 and 500 rpm).
- 3. Extractant concentration (sulfuric acid concentration of 10, 20 and 30wt.% and hydrogen peroxide concentration of 0, 0.05, 0.1 and 0.15 M).
- 4. Pulp density (5, 10, 20 and 30 w/v%).
- 5. Operating temperature (30, 50, 70 and 90° C).

Experimental Procedure Alkali Leaching Stage

In *alkali leaching*, the spent catalyst was ground and sieved. For this study,

two different size fractions of spent catalyst were prepared: (less than 75µm and greater than 75µm) and mixed thoroughly to obtain uniform samples for leaching. The solution was prepared by dissolving a certain amount of sodium carbonate in deionized water to get different concentration of solution. Leaching experiments were carried out by taking 200ml of leachant in 500ml round bottom flask fitted with a seal to avoid evaporation loss. The contents were stirred with mechanical mixer.

After the temperature of the solution reached the desired value, a certain amount of catalyst with different ratios (S/L) was added to the flask and then hydrogen peroxide was added. The addition of H_2O_2 exothermic started the reaction. 10 ml samples were withdrawn at the end of each run and were filtered from residue by vacuum filtration using filtration paper and analyzed for Mo, Co and Al. The analysis was made in Ibn Sina company by using atomic absorption technique. The recovery percentage was calculated by using the following relationship:

$$\%$$
Recovery = $\frac{Amount of metal dissolved}{Total amount of metal} * 100$...(1)

Acid Leaching Stage

In *acid leaching*, the residue of alkali leaching was dried and analysis for Mo, Co and Al. The solution was prepared by adding a certain amount of sulfuric acid to deionized water to get a different concentration of solution. leaching experiments were carried out by taking 200ml of leachant in 500ml round bottom flask fitted with a seal to avoid evaporation loss. The contents were stirred with mechanical mixer.

After the temperature of the solution reached the desired value, a certain amount of catalyst with different ratios (S/L) was added to the flask then hydrogen peroxide was added. 10 ml samples were withdrawn at the end of each run and were filtered from residue by vacuum filtration using filtration paper and analyzed for Co and Al. The analysis was made in Ibn Sina company using atomic absorption technique. The recovery percentage was calculated according to Eq. (1).

Factors Influencing the Experiments Alkali Leaching Stage Effect of Agitation Time

In this set of experiments, the effect of agitation time on Mo extraction was investigated. A sample of solution was taken at the end of each period (30, 60, 90 and 120 min.). Operating conditions (temperature=30°C, agitation speed =300rpm, PD=20%, P.S.=less than 75 μ m, sodium carbonate concentration of 60g/l and 10vol.% hydrogen peroxide concentration) were kept constant.

Effect of Agitation Speed

In this set of experiments, the effect of agitation speed on Mo extraction was investigated. A sample of solution was taken at the end of each run of different speeds (100, 300 and 500 to determine the rpm) metals concentration. Agitation time was fixed from the previous experiments. variables Other such as temperature=30°C, PD=20%, P.S.=less than 75um. sodium carbonate concentration of 60g/l and 10vol.% hydrogen peroxide concentration were kept constant.

Effect of Extractant Concentration

In this set of experiments, the effect of extractant concentration on Mo extraction was investigated. A sample of solution was taken at the end of each run of a different concentration of sodium carbonate (40, 60, 80 and 120 g/l) at fixed hydrogen peroxide concentration (5, 7, 10 and 12 vol.%). From previous sections the agitation time and agitation speed were fixed. Other variables such as operating temperature $=30^{\circ}$ C, PD=20%,and P.S.=less than 75µm were kept constant.

Effect of Pulp Density

In this set of experiments, the effect of pulp density on extraction of Mo was investigated. Different values of pulp density were tried (5, 10, 20 and 30 w/v%). The agitation time, agitation speed, sodium carbonate and hydrogen peroxide concentration were fixed from previous sections and other variables such as temperature $=30^{\circ}$ C and P.S.= -75µm were kept constant.

Effect of Temperature

The effect of temperature on extraction of Mo was investigated. The solution was heated to different temperatures (30, 50, 70 and 90°C). The agitation time, agitation speed, pulp density, sodium carbonate and hydrogen peroxide concentration were fixed from previous sections while particle size was kept constant less than 75 μ m.

Effect of Particle Size

The effect of particle size on extraction of Mo was investigated. Particle size of catalyst less than 75 μ m or (>75 μ m) was used. The agitation speed, pulp density, temperature, sodium carbonate and hydrogen peroxide concentration were fixed from previous sections.

Acid Leaching Stage Effect of Agitation Time

In this set of experiments, the effect of agitation time on Co and Al extraction was investigated. A sample of solution was taken at the end of each period (30, 60, 90 and 120 min.). Operating conditions (temperature=30°C, agitation speed =300rpm, PD=20%, sulfuric acid concentration of 10 wt.% and without hydrogen peroxide) were kept constant.

Effect of Agitation Speed

In this set of experiments, the effect of agitation speed on Co and Al extraction was investigated. A sample of solution was taken at end of each run of different speeds (100, 300 and 500 rpm) to determine the metals concentration. Agitation time was fixed from the previous experiments. Other variables such as temperature=30°C, PD=20%, sulfuric acid concentration of 10wt.% and without hydrogen peroxide were kept constant.

Effect of Extractant Concentration

In this set of experiments, the effect of extractant concentration on extraction of Co and A1 was investigated. A sample of solution was taken at the end of each run of a different concentration of sulfuric acid (10, 20 and 30 wt.%) at fixed hydrogen peroxide concentration (0, 0.05, 0.1and 0.15 M). From previous sections the agitation time and agitation speed were fixed. Other variables such as operating temperature $=30^{\circ}C$ and PD=20% were kept constant.

Effect of Pulp Density

In this set of experiments, the effect of pulp density on extraction of Co and Al was investigated. Different values of pulp density were tried (5, 10, 20 and 30 w/v%). The agitation time, agitation speed, sulfuric acid and hydrogen peroxide concentration were fixed from previous sections and temperature of 30° C was kept constant.

Effect of Temperature

The effect of temperature on extraction of Co and Al was investigated. The solution was heated to different temperatures (30, 50, 70 and 90°C). The agitation time, agitation speed, pulp density, sulfuric acid and hydrogen peroxide concentration were fixed from previous sections.

Results and Discussion Catalyst Characterizations

Powder XRD analysis was performed on the deoiled sample in order to detect metal foulants such as sulfur, argon, titanium, iron and zinc that block the pore mouths and retarding the access of the reactants to the active sites of the catalyst with pores and identify different crystalline phases present in the catalyst. XRD pattern of spent $CoMo/\gamma - Al_2O_3$ catalyst is given in Fig. (2). The analysis was done in the Ministry of Science and Technology.



Fig. 2, X-ray parttern of spent CoMo/ γ -Al₂O₃ catalyst

Alkali Leaching Stage Effect of Agitation Time

The obtained results are plotted in Figs. (3-6) and Fig. 6 illustrate the effect of operating conditions on Mo, Co and Al. It is obvious that the extraction efficiency of Mo increased as the agitation time was increased to a certain point where it remained

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constant. The highest extraction efficiency of 56.37% for Mo was obtained after 90 minutes because extraction processes proceed until equilibrium is reached. Therefore, further increase in the time of experiment had no significant influence on the extraction. The results of Angelidis et al (1995) [6] had also the same trend in selective dissolution of Mo, Co and Ni from diesel and naphtha spent hydrodesulfurization catalyst and in agreement with Shariat et al (2001) [19] in technical note optimizing conditions for hydrometallurgical production of purified molybdenum trioxide from roasted molybdenite of Sarcheshmeh. Co and Al extraction was less than 1% at these operating conditions due to acidity.



Fig. 3, Effect of agitation time on Mo extraction



Fig. 4, Effect of agitation time on Co extraction



Fig. 5, Effect of agitation time on Al extraction



Fig. 6, Effect of agitation time on metals extraction

Effect of Agitation Speed

As seen from Figs. (7-10), the stirring speed shows a small effect on the dissolution of metals within the range used. When the leaching time is 90 min, the stirring speed of 300 rpm gave the best extraction efficiency of Mo (Mo=56.37%). This indicates that the diffusion of the reactants from the solution towards the surface of a catalyst particle and the products away from the surface of the particle was fast and hence increasing agitation speed did not control the leaching rate within the range of the stirring speeds tested. All subsequent experiments were carried out at a stirring speed of 300 rpm. Similar results were observed by Mulak et al (2006) [1]; David (2007) [23] and Qi-ming et al (2009) [24].



Fig. 7, Effect of agitation speed on Mo extraction



Fig. 8, Effect of agitation speed on Co extraction



Fig. 9, Effect of agitation speed on Al extraction



Fig. 10, Effect of agitation speed on metals extraction

Effect of Extractant Concentration

As shown in Fig. (11), a maximum efficiency of Mo extraction was achieved around 60g/L Na₂CO₃ for 90 minutes and decreased gradually thereafter. Where as, the percentage extraction of Co and Al was not significant when compared with Mo extraction as shown in Figs. (12-14).

The effect of H₂O₂ addition varied in values of (5, 7, 10 and 12vol.%) at a constant concentration of Na₂CO₃ (60 g/L). The results are shown in Figs. (15-18). The results showed a gradual decrease of Mo leaching efficiency above 10 vol.% H₂O₂. Whereas, the percentage extraction of Co and Al increased gradually, which is due to the acidic nature of the leach solution (decrease of pH). It was observed that, a particular concentration of Na₂CO₃ and H_2O_2 had a significant effect on maximum Mo solubilization. At higher concentrations of either Na₂CO₃ or H_2O_2 , a reaction with each other probably occurs forming sodium percarbonate and thereby decreases the availability of reactants to react with the catalyst, which causes a decrease in Mo leaching efficiency as mentioned by Park et al (2006b) [25].

Leaching of spent catalyst with Na_2CO_3 in the presence of oxidizing agent such as H₂O₂ involves oxidation of sulphides to sulphate, which is exothermic, and the reaction of Mo with sodium carbonate forming sodium molybdate. The role of oxidant is to break the MoS₂ complex and convert metal and S to the maximum oxidized state and oxidize the oil and free carbon to CO_2 and H_2O . Whereas, the role of Na₂CO₃ is to react with the liberated Mo forming soluble sodium molybdate, neutralization of SO₃ produced during oxidation to soluble sodium sulphate, and thereby avoiding environmental problems. The reaction of molybdenum content of the spent catalyst in presence of Na₂CO₃ and

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 H_2O_2 can be represented by Equation [25]:

 $MoS_{2}+3Na_{2}CO_{3}+H_{2}O_{2}+4O_{2}$ $\rightarrow Na_{2}MoO_{4}+2Na_{2}SO_{4} +H_{2}O + 3CO_{2}$



Fig. 11, Effect of sodium carbonate on Mo extraction



Fig. 12, Effect of sodium carbonate on Co extraction



Fig. 13, Effect of sodium carbonate on Al extraction



Fig. 14, Effect of sodium carbonate on metals extraction



Fig. 15, Effect of hydrogen peroxide on Mo extraction



Fig. 16, Effect of hydrogen peroxide on Co extraction



Fig. 17, Effect of hydrogen peroxide on Al extraction



Fig. 18, Effect of hydrogen peroxide on metals extraction

Effect of Pulp Density

By examining Figs. (19-22), it can be seen that a maximum efficiency of Mo extraction was achieved around 20%PD and decreased gradually thereafter with an increase in the pulp density. Similar behavior was observed by Park et al. (2006a) [26] in hydrometallurgical processing and recovery of molybdenum trioxide from spent catalyst. A large solid to liquid ratio increases the resistance for diffusion of fresh extractant to the surface of catalyst. Therefore, requires longer time and requires higher extractant concentration as concluded by Shariat et al (2001) [19].



Fig. 19, Effect of pulp density on Mo extraction



Fig. 20, Effect of pulp density on Co extraction



Fig. 21, Effect of pulp density on Al extraction



Fig. 22, Effect of pulp density on metals extraction

Effect of Temperature

As temperature increases from 30 to 90°C, the Mo extraction efficiency after 90 minutes leaching was improved from 56.37% to 75%. This is obvious in Figs. (23-26).

An increase in the extraction efficiency was observed with increasing the temperature. This may be attributed to the following reasons: Extraction of Valuable Metals From Spent Hydrodesulfurization Catalyst By Two Stage Leaching Method

• Increasing leaching temperature will increase the rate of reaction between the solvent and value mineral. It was found that the relation between the rate of reaction and the temperature at which the reaction takes place can be represented by the Arrhenius equation [27].

$$K = A. e^{\left(-\frac{E}{RT}\right)} \qquad \dots (3)$$

Where: K is the reaction rate constant.

• Increasing the temperature will increase the diffusion coefficient, since it also follows an Arrhenius relationship [28].

$$D = D_0 \cdot e^{\left(-\frac{E}{RT}\right)} \qquad \dots \quad (4)$$

Where : D is the diffusion coefficient.

Similar results were observed by Angelidis *et al* (1995) [6] in selective disolution of critical metals from diesel and naphtha hydrodesulfurization catalyst.



Fig. 23, Effect of temperature on Mo extraction



Fig. 24, Effect of temperature on Co extraction



Fig. 25, Effect of temperature on Al extraction



Fig. 26, Effect of temperature on metals extraction

Effect of Particle Size

It can be seen from Figs. (27-29) that the particle size has a significant effect on dissolution of metals. It was found that the highest extraction efficiency was achieved at a catalyst particle size of less than 75 μ m. The Mo extraction efficiency for (<75) and (>75) μ m particle size reaches 75% and 56.46%, for Co efficiency reaches 1.34% and 1.05% and for Al reaches 0.98% and 0.77% after 90 minutes leaching, respectively.

By decreasing the particle size, the contact area between the catalyst particle and the fluid is increased, therefore the reaction rate is increased which is also concluded by Souza *et al* (2007) [29] and Qi-ming *et al* (2009) [24].

Since the acid leaching involved leaching of residue from alkali leaching, particle size was the same.



Fig. 27, Effect of particle size on Mo extraction



Fig. 28, Effect of particle size on Co extraction



Fig. 29, Effect of particle size on Al extraction

Acid Leaching Stage Effect of Agitation Time

Fig. (30) shows increasing of extraction efficiency with increasing the agitation time which was also observed by Anglidis *et al* (1995) [6], Mulak *et al* (2006) [1], David (2007) [23] and Qi-ming *et al* (2009) [24]. Experiments showed that 90 minutes contact time is sufficient enough to reach equilibrium between solid phase and aqueous phase. Only Co and Al seem to dissolve considerably, and

small quantities of remaining Mo were detected in leachate. The quantity of dissolved Al was considerably higher than the one achieved during alkali leaching experiments, since alumina is readily soluble in acids as mentioned by Anglidis *et al* (1995) [6].



Fig. 30, Effect of agitation time on metals extraction

Effect of Agitation Speed

The results presented in Fig. (31), show that the leaching of cobalt and aluminum is independent of the stirring speed. In the study on a spent HDS catalyst leaching with sulfuric acid, Mulak et al (2005) [30] also observed similar behavior of stirring speed on the leaching kinetics. This indicates that the external diffusion of H₂SO₄ and CoSO₄ between the fluid and the surface of particles is fast due to small particle size, and hence does not control the dissolution of metals within the investigated range of stirring speed. subsequent experiments All were carried out at a stirring speed of 300 rpm in order to assure the invariance of this parameter.



Fig. 31, Effect of agitation speed on metals extraction

Effect of Extractant Concentration

As shown in Fig. (32), the effect of sulfuric acid concentration on cobalt and aluminum extraction efficiency increases with increasing the sulfuric acid concentration. For 10 wt.% concentration, there is virtually an acid deficiency effect. Nevertheless, there is no beneficial effect of increasing acid concentration more than 20%. However, after elapsing a certain time, namely, 90 minutes, the metals extraction efficiency practically remains constant. The increase in the extraction of Co and Al may be due to the following reasons:

- The high penetrative of acid through the solid particle.
- The high reaction rate between the acid and metals.

These results are in agreement with Angelidies et al (1995) [6] and Qiming et al (2009) [24]. The cobalt extraction efficiencies after 90 minutes extraction with 10%, 20% and 30% sulfuric acid are found to be 35.93%, 40.43% and 40.43%, respectively and for aluminum extraction efficiencies are found to be 6.53%, 8.84% and 8.86%, respectively.

The influence of H_2O_2 concentration on the extraction of cobalt and aluminum from the spent CoMo/y-Al₂O₃ catalyst was determined by varying the initial concentration of H_2O_2 from 0.0 to 0.15M at 30°C in 20wt.% H₂SO₄ solution. Fig. (33) shows summarized metals extraction after 90 minutes extraction. Generally, the extraction of Co and Al gradually increased up to $0.1M H_2O_2$ and then remained constant. This behavior may be explained by the fact that as the acid concentration in the medium increases, the apparent rate of solid product (CoSO₄) increases, and the internal diffusion resistance of H₂SO₄ through the layer is enhanced. product Consequently, the dissolution process slows down. The extraction of Al was

practically not affected by the concentration of H₂O₂ within the whole range. The highest extraction of cobalt (48%) and aluminum (8.8%) was observed in 20wt.% H₂SO₄ solution with 0.1M H_2O_2 at 30°C during the 90 minutes leaching. Hydrogen peroxide was added to H₂SO₄ solution to help metals dissolution from spent catalyst. These results were in agreement with results of Mulak *et al* (2006) [1] in preliminary results of Ni, Mo, Al and V leaching from a spent NiMo/ γ -Al₂O₃ catalyst and David (2007) [23] on extraction of valuable metals based on Cu, Ni $/\gamma$ -Al₂O₃.



Fig. 32, Effect of sulfuric acid concentration on metals extraction



Fig. 33, Effect of hydrogen peroxide concentration on metals extraction

Effect of Pulp Density

Fig. (34) indicates that no significant variations were observed on the dissolution of the valuable metals with

the increase in the solid to liquid ratio. Thus 20%PD was the best value for metals extraction. This trend might indicate that equilibrium leaching conditions were reached even by using the lower solid to liquid ratio. These results are in agreement with David (2007) [23].



Fig. 34, Effect of pulp density on metals extraction

Effect of Temperature

The temperature increas from 30 to 90°C caused the cobalt extraction efficiency after 90 minutes leaching to be improved from 48% to 93.2% and for aluminum from 8.8% to 54.3% according to Arrhenius Eq. (3) and Eq. (4) as shown in Fig. (35). These results are in agreement with Angelidis *et al* (1995) [6] and Qi-ming *et al* (2009) [24].



Fig. 35, Effect of temperature on metals extraction

Conclusions

For the present investigation and operating conditions used, it may be concluded that:

- ✤ For alkali leaching:
- 1. Alkali leaching for Mo extraction by using Na_2CO_3 and H_2O_2 , the equilibrium was reached after 90 minutes of contact time.
- 2. Agitation speed had small effect on the extraction of Mo and 300rpm gave the best result.
- 3. The rate of Mo extraction increased with increasing in Na_2CO_3 and H_2O_2 concentrations to 60 g/l and 10vol.%, respectively and, thereafter, decreased gradually.
- 4. A maximum efficiency of Mo was achieved around 20% PD and, thereafter, decreased gradually.
- 5. The results indicate that increasing the temperature from 30 to 90 °C lead to an increasing in the Mo extraction from 69.12% to 75%.
- 6. The rate of Mo extraction increased with decreasing the particle size. The particle size which is less than 75μ m gave the best extraction than the particle size which greater than 75μ m.
- 7. The results showed that the process controlled by both mass and reaction mechanisum.
- ✤ For acid leaching:
- 1. Acid leaching for extraction of Co and Al by using H₂SO₄ and H₂O₂, the equilibrium was reached after 90 minutes of contact time.
- 2. Agitation speed had small effect on Co and Al extraction and 300rpm gave the best result.
- 3. The rate of Co and Al extraction increased with increasing H_2SO_4 and H_2O_2 concentrations up to 40.43% and 48% for cobalt and up to 8.84% and 8.8% for aluminum and remained constant thereafter.
- 4. It was found that no significant variations on the dissolution of Co and Al with the increase in pulp density.

- 5. The rate of Co and Al extraction increased with increasing the temperature up to 90° C.
- 6. The results showed that the process was controlled by both mass and reaction mechanisum.

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