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OPTIMIZATION OF Hg-FLOTATION FROM THE AGH-DARREH GOLD MINE TAILINGS

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ABSTRACT

The application of the flotation process for removing of Hg from the tailings of the Agh-Darreh gold processing plant was investigated using a software-based design of experiments. Six main parameters: pH, solid content, collector concentration, depressant concentration, activator concentration, and collector type, were considered in a central composite response surface design. The results showed that the most effective parameters are pH, solid content, collector concentration, collector type, depressant concentration, and activator concentration, respectively. In optimum conditions 68.99 % Hg can be removed when the collector is PAX, solid content of 31 % and collector, depressant and activator concentrations are 92.74, 57.13 and 17.81 ppm respectively. It can be concluded that the froth flotation is a useful method for removing Hg from the tailings of the Agh-Darreh gold processing plant.

Keywords: gold ore, response surface methodology, flotation, mercury, optimization. Agh-Darreh.

INTRODUCTION

Mercury sulfide, or cinnabar (HgS), is the most common form of mercury mineralization both in mercury mines and also in precious metal deposits [1, 2]. Cyanide leaching is used to remove the gold and silver from the ore and results in the dissolution of some mercury as a cyanide-mercury complex [3 - 5]. During the cyanidation process, generally, only 10 % - 40 % of mercury in the ore is extracted along with the gold and silver, although the extent of mercury extraction is highly dependent on the concentration of cyanide in solution and the mineralogy of the mercury [6]. Mercury forms considerably strong cyano complexes, $Hg(CN)_3^-$ and $Hg(CN)_4^{-2}$. The mercury cyano-complexes are much stronger and more persistent in tailings solutions and the environment [7]. Mercury concentrations from 2 to 495 ng g^{-1} were determined in the tailings [8].

Mercury is generally considered to be the most toxic of the heavy metals in the environment [9, 10]. Recovery of mercury from mercury-rich tailings in artisanal and small-scale gold mining by cyanidation processes was studied. The results showed that at 10 g ton^{-1} of cyanide, approximately 42 % of the mercury was leached, whereas all gold was solubilized [11].

Cinnabar can be easily recovered by flotation using pine oil and a collector such as a xanthate [12, 13]. Floatability of cinnabar is reduced due to surface oxidation when it is exposed to the atmosphere after crushing. This problem can be eliminated by adding copper sulfate and lead nitrate or acetate. The best pH for cinnabar flotation is in the range of 6.5 to 8.5. Cinnabar and stibnite can be floated with long-chain xanthate collectors and an alcohol frother, with cumulative recoveries of 97.8 % and 94.7 %, respectively [14].

In this research recovery of mercury from the tailing of Agh-Darreh gold ore mine was investigated. Agh-Darreh mine and gold processing plant is located 32 km from Takab city in western Azerbaijan province, Iran. The grade of gold ore is 3 ppm. Cyanide leaching is used to process gold in the Agh-Darreh mine. The ore from the mine is crushed and milled (AG mill) in a closed circuit (P_{80} = 75 μm). Only 10 % of the total mercury (85 - 150 ppm) is recovered since just 20 % of the total mercury is leached by cyanide in the leaching circuit and about 50 % of the dissolved mercury is adsorbed onto activated

Table 1. Particle size distribution and Hg distribution in the sample.

Size (μm)	Mass remained (%)	Hg (ppm)	Hg distribution (%)
212	0.54	61	0.31
150	0.88	55	0.45
106	3.35	46	1.43
75	4.91	64	2.9
53	8.99	87	7.27
38	6.91	114	7.33
-38	74.42	116	80.29

carbon. Unleached mercury, which comprises mainly cinnabar and metacinnabar, goes to the tailings dam. In this research, flotation experiments were conducted on the leach tailings from the Agh-Darreh gold processing plant in order to recover the mercury. The effects of flotation parameters such as pH, solid content, collector concentration, activator concentration, depressant concentration and collector type on mercury flotation were studied.

EXPERIMENTAL

Sampling

The sample used in this research was taken from the last leaching tank of the plant. The sample was taken for six days to ensure that a representative sample had been collected.

Materials characterization

Table 1 shows the particle size distribution and Hg distribution of the sample. Data from Table 1 shows that the sample is fine and 74 % finer than 38 μm and approximately 80 % of Hg content is in the size fraction of 38 μm. Table 2 shows the chemical compositions of the representative sample, determined by X-ray fluorescence (XRF, Philips X Unique II) and atomic absorption spectrophotometry (AAS, Agilent model AA240FS). As observed from Table 2, the sample was mainly composed of SiO_2, CaO, Fe_2O_3, MgO, Al_2O_3, Na_2O, and Fe_2O_3, respectively, and the content of Hg is 90.4 ppm.

The XRD analyses showed that quartz, calcite, smectite, illite, dolomite, barite, goethite, kaolinite, iron arsenate dehydrate, jarosite, sepiolite, and pyrite are the minerals present in the sample in order of abundance.

For the investigation of mineral liberation, four polished section from +150μm, -150+100μm, -100+40μm, -40+10μm and -10μm size fractions were provided and studied by Zeiss Axioplan2 research optical microscope. The Mineralogical analysis revealed, approximately 37 % of Hg minerals are associated with other minerals, 43 % are liberated as cinnabar and meta-cinnabar and 20 % are as native Hg and HgCN salts. 95 % of liberated Hg minerals are less than -10 μm.

Flotation experiments

Flotation experiments were carried out in a 5500 cm^3 conventional laboratory cell (Denver model, agitation rate of 1200 rpm). The suspension was added to the flotation cell and conditioned for 5 min. The pH was adjusted to the desired value (6 - 11) by adding $Ca(OH)_2$ or H_2SO_4. A mixture of starch and sodium silicate as a depressant for clay, silicate and, iron oxide minerals and copper sulfate as an activator (to reduce the effect of surface oxidation) for cinnabar was then added and the suspension was conditioned for 2 min. The potassium amyl xanthate (PAX) or potassium ethyl xanthate (PEX) was added as the collector and the suspension was conditioned for 2 min. Finally, MIBC (40 g ton^{-1}) was added as the frother then after 3 min air was delivered to the cell for a total of 8 min and the concentrate which was

Table 2. Chemical composition of the representative sample.

Composition	Content
Hg*	90.4ppm
SiO_2	36.99%
Al_2O_3	2.25%
Fe_2O_3	6.5%
CaO	10.88%
MgO	2.5%
Na_2O	0.91%
*Hg was determined by AAS	

floated and remained in the suspension was collected, filtered and dried in a well-ventilated oven at 120ºC.

Experiment's plan was designed by DX7 software. Five variable numeric parameters (pH, solid content, collector concentration, depressant concentration, activator concentration) and collector type as categorical parameter were considered in this investigation and consequently 54 experiments with central composite design (CCD) were carried out. Response surface methodology (RSM) and central composite rotatable design (CCRD) were used to model and optimize the influence of the parameters affecting cinnabar flotation process [15, 16].

RESULTS AND DISCUSSION

The numeric variable parameters and the coded/actual values are given in Table 3. The plan and results of the experiments are given in Table 4.

The results were inserted to "Design Expert (DX7)" software and a model was chosen and fitted to the results. The response of the quadratic polynomial is described in the following equation in terms of coded factors:

For F: PAX

$$\begin{aligned} Hg\ Recovery = (&-57.26 + 10.106\,A + 2.104\,B \\ &+ 1.072\,C + 0.094\,D - 0.587\,E \\ &- 0.151\,AC - 0.042\,BC \\ &+ 0.00009\,BD + 0.0006\,CD \\ &+ 0.004\,CE - 0.011\,DE \\ &- 0.128\,A^2 - 0.001\,B^2 \\ &- 0.00003\,C^2 + 0.0003\,D^2 \\ &+ 0.009\,E^2)^2 \end{aligned} \tag{1}$$

For F: PEX

$$\begin{aligned} Hg\ Recovery = (&-48.597 + 10.319\,A + 1.129\,B \\ &+ 1.152\,C - 0.166\,D + 1.21\,E \\ &- 0.151\,AC - 0.042\,BC + 0.003\,BD \\ &+ 0.001\,CD + 0.001\,CE - 0.012\,DE \\ &- 0.128\,A^2 + 0.014\,B^2 - 0.0008\,C^2 \\ &+ 0.0003\,D^2 - 0.004\,E^2)^2 \end{aligned} \tag{2}$$

The results of the analysis of variance (ANOVA) consisting of the p-value, the sum of square, mean square, F-value and degree freedom (df) are shown in Table 5. The results of the analysis of variance (ANOVA) showed that the prediction model of the Hg recovery is significant because the p-value of the both model is under 0.0001 and lack of fit is not significant. The influence of A, B, C and F on the Hg recovery are significant (p-value < 0.05) but the influence of D and E are not significant (p-value > 0.05). Also, many interactions between variables (AC, BC, BD, CD, CE, DE, DF and EF) are significant. The relationship between the predicted values and the actual values for Hg recovery is given in Fig. 1.

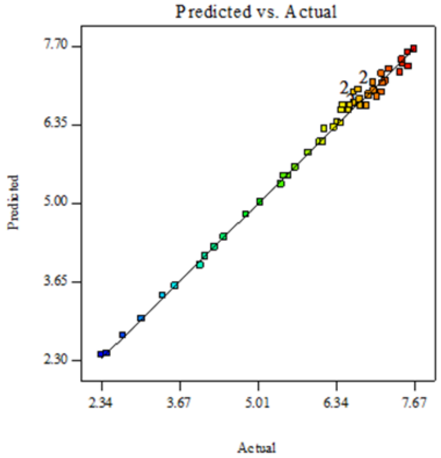

Fig. 1. Relationship between predicted and actual recovery (observed values for Hg).

Table 3. Coded and actual levels of the numeric variables parameters used in the experimental design.

Variable	Symbol	Coded variable level				
		-2	-1	0	1	2
pH	A	6	7.3	8.5	9.8	11
Solid content (%)	B	20	25	30	35	40
Collector concentration (ppm)	C	10	32.5	55	77.5	100
Depressant concentration (ppm)	D	0	25	50	75	100
Activator concentration (ppm)	E	0	5	10	15	20

Table 4. The plan of experiments with experimental results.

Run	Block	pH	Activator conc. (ppm)	Depressant conc. (ppm)	Collector conc. (ppm)	Solid content (%)	Type of collector	Hg Recovery (%)
1	Block 1	6	20	100	100	20	PEX	23.1
2	Block 1	11	20	100	100	0	PEX	29.2
3	Block 1	11	40	100	0	0	PEX	25.46
4	Block 1	11	40	10	100	0	PAX	16.2
5	Block 1	6	20	100	100	20	PAX	39.7
6	Block 1	8.5	30	55	50	10	PEX	43.31
7	Block 1	8.5	30	55	50	10	PEX	45.52
8	Block 1	8.5	30	55	50	10	PEX	47.13
9	Block 1	11	20	100	0	20	PEX	31.86
10	Block 1	8.5	30	55	50	10	PEX	45.52
11	Block 1	6	40	100	100	0	PEX	19.6
12	Block 1	11	40	10	0	20	PAX	30.57
13	Block 1	11	20	100	100	0	PAX	52.5
14	Block 1	11	40	10	100	0	PEX	11.46
15	Block 1	11	40	100	0	0	PAX	36.78
16	Block 1	11	20	10	100	20	PAX	40.45
17	Block 1	6	40	100	0	20	PEX	18.19
18	Block 1	11	40	10	0	20	PEX	5.46
19	Block 1	8.5	30	55	50	10	PAX	44.12
20	Block 1	8.5	30	55	50	10	PAX	44.12
21	Block 1	6	20	10	0	0	PEX	9.13
22	Block 1	8.5	30	55	50	10	PAX	50.6
23	Block 1	6	20	10	0	0	PAX	29.78
24	Block 1	11	20	100	0	20	PAX	50.65
25	Block 1	8.5	30	55	50	10	PAX	49.18
26	Block 1	6	40	100	100	0	PAX	34.43
27	Block 1	6	40	10	100	20	PAX	16.87
28	Block 1	6	40	10	100	20	PEX	5.93
29	Block 1	11	20	10	100	20	PEX	7.32
30	Block 1	6	40	100	0	20	PAX	12.94
31	Block 2	8.5	30	55	50	15	PAX	49.56
32	Block 2	8.5	25	55	50	10	PAX	55.24
33	Block 2	8.5	30	55	75	10	PEX	42.98
34	Block 2	9.75	30	55	50	10	PAX	51.52
35	Block 2	8.5	30	55	50	10	PEX	47.49
36	Block 2	8.5	30	32.5	50	10	PEX	41.73
37	Block 2	8.5	30	55	25	10	PAX	48.68
38	Block 2	8.5	30	55	75	10	PAX	57.23
39	Block 2	8.5	35	55	50	10	PAX	45.51
40	Block 2	8.5	30	55	50	10	PAX	48.6
41	Block 2	8.5	30	55	50	15	PEX	50.89
42	Block 2	8.5	30	77.5	50	10	PAX	44.16
43	Block 2	8.5	30	55	25	10	PEX	55.7
44	Block 2	8.5	30	55	50	10	PAX	48.6
45	Block 2	8.5	35	55	50	10	PEX	45.1
46	Block 2	8.5	30	32.5	50	10	PAX	57.33
47	Block 2	8.5	30	77.5	50	10	PEX	37.42
48	Block 2	7.25	30	55	50	10	PEX	41.29
49	Block 2	8.5	30	55	50	5	PEX	37.71
50	Block 2	8.5	25	55	50	10	PEX	55.8

Table 5. Analysis of variance (ANOVA) of developed models for Hg recovery.

Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)	Not
Model	73.08	30	2.44	84.54	< 0.0001	significant
A: pH	0.80	1	0.80	27.66	< 0.0001	significant
B: Solid content	0.73	1	0.73	25.30	< 0.0001	significant
C: Collector concentration	0.40	1	0.40	14.00	0.0011	significant
D: Depressant concentration	0.039	1	0.039	1.34	0.2597	not significant
E: Activator concentration	0.001	1	0.001	0.041	0.8422	not significant
F: Type of collector	0.40	1	0.40	13.88	0.0012	significant
AC	1.59	1	1.59	55.19	< 0.0001	significant
BC	1.65	1	1.65	57.23	< 0.0001	significant
BD	0.92	1	0.92	31.87	< 0.0001	significant
CD	2.19	1	2.19	75.96	< 0.0001	significant
CE	1.39	1	1.39	48.35	< 0.0001	significant
DE	2.13	1	2.13	73.84	< 0.0001	significant
DF	1.08	1	1.08	37.49	< 0.0001	significant
EF	1.20	1	1.20	41.56	< 0.0001	significant
A^2	0.24	1	0.24	8.38	0.0084	significant
B^2	0.11	1	0.11	3.76	0.0653	significant
C^2	0.32	1	0.32	11.11	0.0030	significant
D^2	0.20	1	0.20	6.77	0.0163	significant
ABC	2.28	1	2.28	79.12	< 0.0001	significant
ADF	0.57	1	0.57	19.69	0.0002	significant
AEF	2.81	1	2.81	97.67	< 0.0001	significant
BCF	1.34	1	1.34	46.47	< 0.0001	significant
BDF	1.39	1	1.39	48.18	< 0.0001	significant
BEF	0.96	1	0.96	33.30	< 0.0001	significant
CDF	0.93	1	0.93	32.26	< 0.0001	significant
CEF	0.50	1	0.50	17.31	0.0004	significant
DEF	1.72	1	1.72	59.53	< 0.0001	significant
B^2F	0.23	1	0.23	7.93	0.0101	significant
C^2F	0.28	1	0.28	9.70	0.0050	significant
E^2F	0.17	1	0.17	5.74	0.0256	significant
Residual	0.63	22	0.029	-	-	-
Lack of Fit	0.41	14	0.029	1.05	0.4911	not significant
Pure Error	0.22	8	0.028	-	-	-
Cor Total	108.49	53	-	-	-	-

For a good fit of a model, R^2 and Adjusted R^2 should be at least 0.80 and predicted R^2 should be at least 0.5 [17]. R^2, adjusted R^2 and predicted R^2 were found to be 0.9914, 0.9797 and 0.5926, respectively. The value of R^2 shows that there is an acceptable relationship between the predicted and actual values; therefore, the model fitted is significant.

Effects of parameters on Hg recovery

The effects of the variable parameters on the Hg recovery are illustrated in Fig. 2.

The results showed that the most effective parameters are pH, solid content, collector concentration, collector type, depressant concentration, and activator concentration, respectively. The Hg recovery increased with increasing the pH from 6 to 9, with further increase in the pH from 9 to 11 the Hg recovery decreased. Previous works demonstrated that maximum recovery of cinnabar by xanthate obtains at pH 8.5 - 10.5 [18, 19].

The Hg recovery decreased with increasing in solid content, collector concentration, and activator concentration, while increasing in depressant concentration

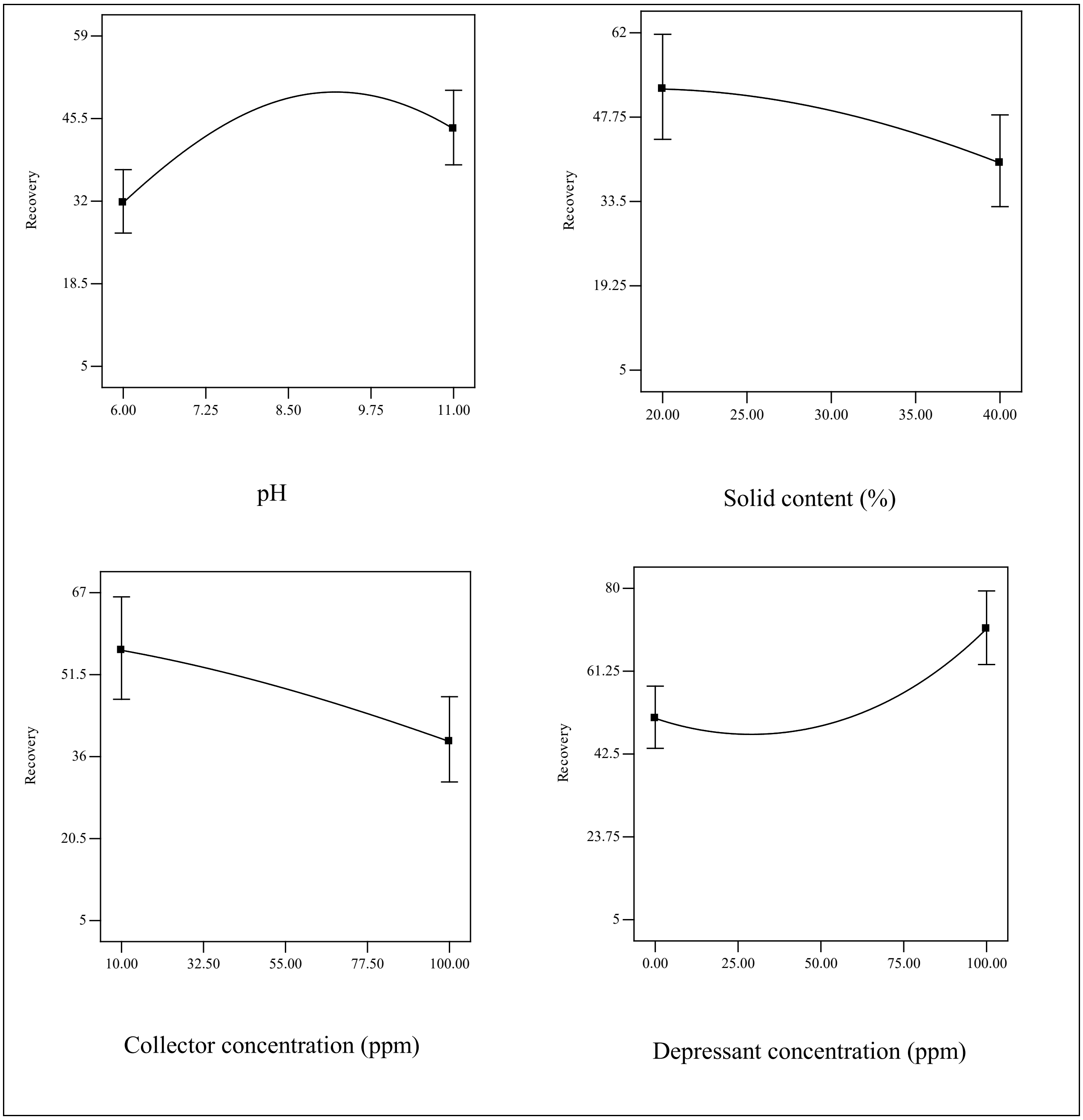

Fig. 2. Effects of variable parameters on Hg recovery (except where the effect of the collector type has been investigated, in other cases the collector was PAX and other variables are held at center level).

increased the Hg recovery. Increasing solid content causes over-crowding of particles, and this hinders effective dispersion and flotation and affects the selectivity. Decreasing particle dispersion reduces the selective absorption of reagents on the particles and also enhances the recovery of gangue particles by the entrainment and entrapment phenomena [20 - 22]. Increasing the copper sulfate causes the collector to precipitate in the form of copper oxide and thus the Hg recovery is reduced.

PAX collector compares with PEX collector was more efficient and enhanced the Hg recovery. PAX compare to PEX has a longer hydrocarbon chain. Upgrading of ore having finely ground sulfide minerals can be possible with the use of long-chain xanthates, resulting in better flotation recovery by inducing aggregation and hydrophobization of the fine sulfide particles [23 - 28].

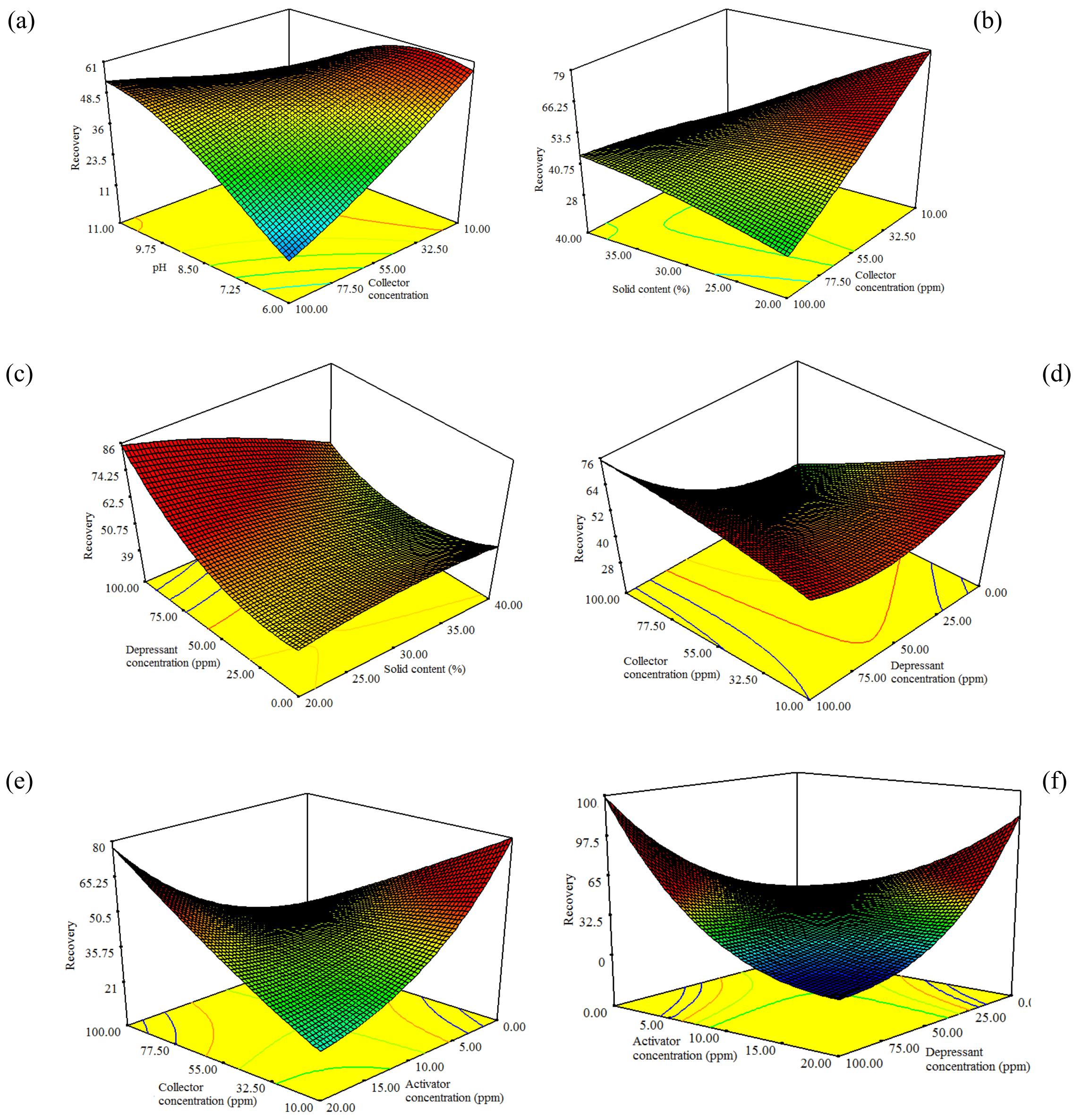

Fig. 3. Interactions of pH and collector concentration (a), solid content and collector concentration (b), solid content and depressant concentration (c), collector concentration and depressant concentration (d), collector concentration and activator concentration (e), activator concentration and depressant concentration (f) on Hg recovery, (collector PAX and other variables are held at center level).

Furthermore, the interactions between variables and their influence on the Hg recovery were investigated. The results showed that there are meaningful interactions between pH and collector concentration, solid content and collector concentration, solid content and depressant concentration, collector concentration and depressant concentration, collector concentration and activator concentration, activator concentration and depressant concentration, depressant concentration and collector type, activator concentration and collector type. These results are given in Fig. 3 and Fig. 4.

Fig. 3(a) shows that at low collector concentration the Hg recovery decreased with increasing pH, but at high collector concentration, the Hg recovery increased with increasing pH. The maximum Hg recovery was achieved at pH = 6 and collector concentration of 10 ppm and the minimum Hg recovery (about 5 %) was achieved at pH = 6 and collector concentration of 100

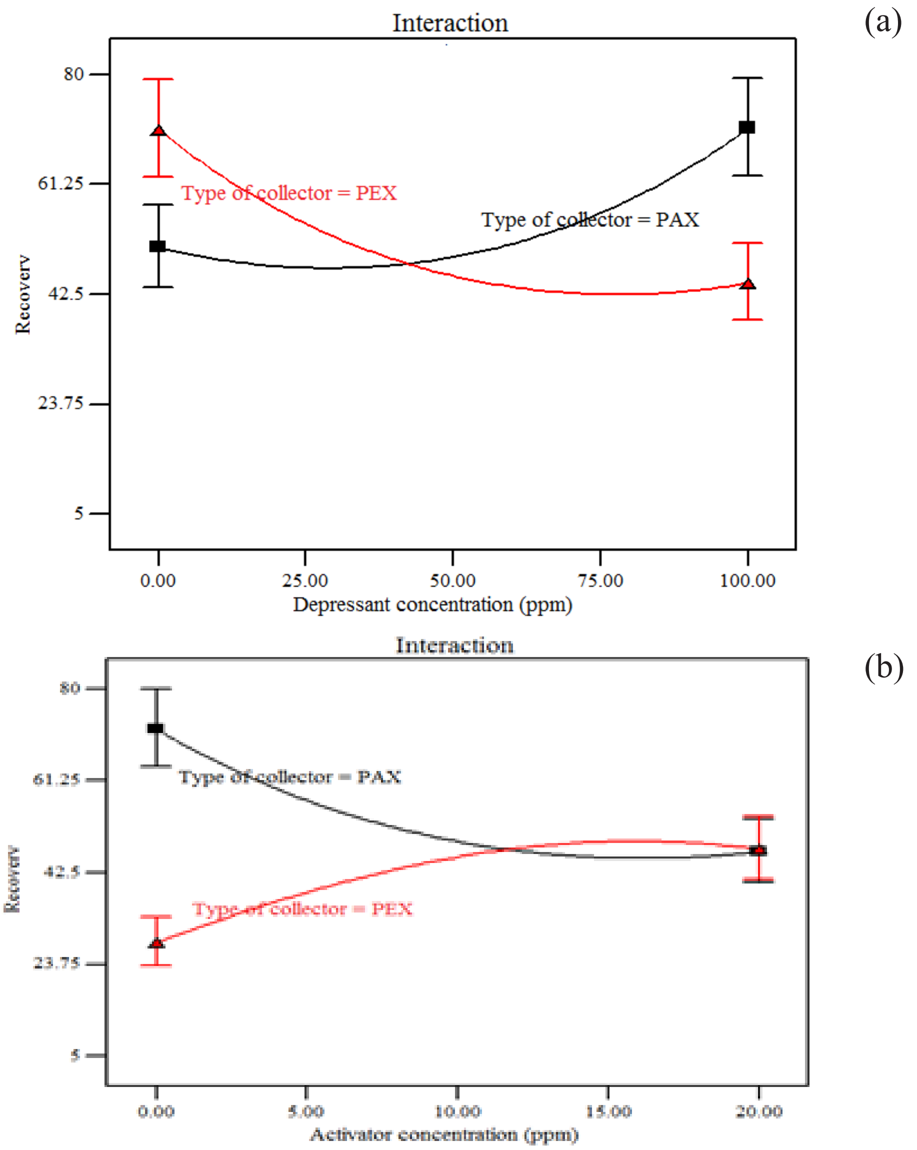

Fig. 4. Interactions of depressant concentration and collector type (a), activator concentration and collector type (b) on Hg recovery (other variables are held at center level).

ppm. Fig. 3(b) shows that at low collector concentration the Hg recovery decreased sharply with increasing solid content but at high collector concentration the Hg recovery increased slightly with increasing solid content. The maximum Hg recovery was achieved at solid content of 20 % and collector concentration of 10 ppm. Fig. 3(c) shows that at high depressant concentration the Hg recovery decreased with increasing solid content but at low depressant concentration the Hg recovery increased slightly with increasing solid content. The maximum Hg recovery was achieved at solid content of 20 % and depressant concentration of 100 ppm. Fig. 3(d) shows that at low depressant concentrations the Hg recovery decreased sharply with increasing collector concentration but at high depressant concentration the Hg recovery is generally high and increasing collector concentration have little effect on the Hg recovery. Fig. 3(e) shows that at low activator concentration the Hg recovery decreased with increasing collector concentration but at high activator concentration, the Hg recovery increased with increasing collector concentration. The maximum recovery can be achieved without activator and collector concentration of 10 ppm or at activator concentration of 20 ppm and collector concentration of 100 ppm. Fig. 3(f) shows that at low activator concentration the Hg recovery increased sharply with increasing depressant

Table 6. Results of process optimization and optimum levels of variables.

pH	Soild wieght (%)	Collector concentration (ppm)	Depressant concentration (ppm)	Activator concentration (ppm)	Type of collector	Recovery (%)	Desirability
9.50	31.00	92.74	57.13	17.81	PAX	68.99	1

Table 7. Confirmation experiments at the predicted optimum conditions.

Experiment Num	pH	Soild wieght (%)	Collector concentration (ppm)	Depressant concentration (ppm)	Activator concentration (ppm)	Type of collector	Predicted Recovery (%)	Experimental Recovery (%)
1	9.50	31.00	92.74	57.13	17.81	PAX	68.99	68.20
2								68.00
3								68.50

concentration while at high activator concentration the Hg recovery decreased with increasing depressant concentration.

Fig. 4(a) shows that when the collector is PAX the Hg recovery increased with increasing depressant concentration, but when the collector is PEX the Hg recovery decreased with increasing depressant concentration. Fig. 4(b) shows that when activator concentration increased from 0 - 10 ppm, the Hg recovery decreased when the collector is PAX and increased when the collector is PEX. Further increasing in activator concentration up to 20 ppm has no significant effect on the Hg recovery and both collector types almost provide the same Hg recovery.

Optimization

Finding an optimum condition of the flotation process with the highest Hg recovery, the lowest consumption of chemical materials was the main object of this investigation. The response surface methodology can be used to find a desirable location in the design space. Variables can be minimum or maximum in this location; also, the economic condition was considered to find a desirable location. The results of the process optimization and optimum levels of variables are demonstrated in Table 6. The data presented in Table 6 indicates that 68.99 % Hg can be recovered when the collector is PAX, solid content of 31 % and collector, depressant and activator concentrations are 92.74, 57.13 and 17.81 ppm, respectively. The desirability of this predicted condition achieved 1.

To confirm the validity of the model, three confirmation experiments were carried out under the predicted optimum conditions (Table 7). The results verify the prediction model and optimum conditions.

CONCLUSIONS

Removing of mercury from the tailings of the Agh-Darreh gold processing plant using flotation process was investigated. The results showed that the most effective parameters are pH, solid content, collector concentration, collector type, depressant concentration and activator concentration, respectively. In optimum conditions 68.99 % Hg can be recovered when the collector is PAX, solid content of 31 % and collector, depressant and activator concentrations are 92.74, 57.13 and 17.81 ppm, respectively. The desirability of optimum condition was approximately 1. As a result, flotation can be a useful method for removing Hg from the tailings of the Agh-Darreh gold processing plant.

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