



Vol. 6, No. 1, Spring 2021, pp. 71-81

دوره ششم، شماره ۱، بهار ۱۴۰۰، صفحه ۷۱ تا ۸۱



نشریه مهندسی منابع معدنی

Journal of Mineral Resources Engineering
(JMRE)

DOI: 10.30479/jmre.2021.14041.1439

Research Paper

Comparison of the Effect of Tap Water and Process Water on the Galena and Sphalerite Minerals Flotation

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(Received: 31 Aug. 2020, Accepted: 16 Dec. 2020)

Abstract

In this study, the effect of recycled water on the grade and recovery of lead and zinc sulfide flotation in comparison with Tap water has been investigated. The ore sample was prepared from Anguran lead and zinc mine, and the process water sample was prepared from KZG (Kimia Zanzan Gostaran) lead and zinc flotation plant. The results showed that when process water is used compared to Tap water, the recovery and grade of Galena and Sphalerite decreased. In Galena flotation, the recovery using Tap water was 3% higher than process water, and the grade with Tap water was significantly higher (8%). In Sphalerite flotation with Tap water, the recovery and grade were significantly increased (11% of recovery and 10% of grade). Investigation of the oxides in Galena and Sphalerite concentrates revealed that the number of oxides that were non-selective activated in the presence of process water is higher. This result is due to flotation reactant residues in these waters and their Precipitation on Galena and Sphalerite ores and non-selective activation of other minerals.

Keywords

Flotation, Process water flotation, Galena and Sphalerite flotation.

1- INTRODUCTION

Water is a critical factor in mines and mineral processing industries. On average, in a lack of recycling the water in a processing plant, water consumption is in the range of 1.9-3 m³/ton of ore processed [1]. Most mineral processing sections, such as crushing, flotation, gravity concentration like the heavy-media separation method, and hydrometallurgical processes, consume a large volume of water. With the increasing demand for water and lack of access to freshwater, water recycling in processing plants is vital and necessary. However, according to the available data, the chemical composition of process water is different from the freshwater and may have some effects on the concentration process.

According to previous research, lack of access to the surface of valuable minerals due to ions and process water complexes, activators, and collectors compete with other species for adsorption on valuable minerals. Loss of reactants due to their precipitation, unwanted reactions, and adsorption on the surface of useful minerals and covering the surface of valuable minerals and preventing them' surface contact with reactants, leading to the recovery decreasing. Non-selective coatings on the surface of valuable minerals and gangue, Ineffective separation of gangue from the valuable mineral, and ineffective activation of the valuable mineral decrease grade factors [2]. Reusing processed water is a better alternative way to decrease the demand for freshwater. Process water or low-quality water can have undesirable effects on the quality of process water due to the accumulation of certain chemical compounds [3]. The use of process water in flotation is having a significant impact on selectivity and recovery. Process water may contain some of the reactant residues and their oxidation compounds, metal ions, alkali metal ions [4]. Water closed circuits in flotation plants lead to high electrolyte concentrations in processed water [5]. The presence of complexes and ions in process water causes non-selective oxidation and reduction in minerals. The released ions by reacting with minerals lead to non-selective activation or inhibition of them. Meanwhile, free sulfur particles in the pulp have a significant role in hydrophobe other minerals [6]. The reason for high foam in flotation with process

water is probably due to the presence of ions and residual complexes of soluble reactants (such as sulfur, sulfate compounds, and water-soluble salts) [7]. Detergents, organic material, dissolved solids, reactants, and dissolved oxygen in process water affect the gangue hydrophobicity. therefore, gangue floats with valuable minerals [8]. Water-soluble sulfur in the form of sulfate compounds in more than a concentration of 2000 mg/l results in the formation of lead sulfate, a hydrophilic and inactivated compound [9].

Water has a direct impact on processing and its effects on flotation should be investigated. According to earlier research and studies, a few research has been done on the effect of recycled water of lead and zinc on the grade and recovery of both Galena and Sphalerite flotation, especially Galena and Sphalerite of Anguran mine. In this study, after Tap water, process water, and minerals have been identified, the Comparative effect of process water and Tap water on the grade and recovery of Galena and Sphalerite flotation has been investigated. Besides, water pollution from both ionic and environmental aspects has been studied to analyze the environmental pollution of this affluent and the necessity for water treatment. Ionic contamination origin of recycled water was studied. Also, by comparing the concentrates of recycled water and tap water, the reason for the different results has been investigated and analyzed. To improve the results of flotation grade and recovery, solving the problem of environmental pollution and water shortage, which are three water issues in flotation, a solution was proposed.

2- MATERIAL AND METHODS

2-1- Materials

100 kg mixture ore of Galena and Sphalerite of Anguran Lead/Zinc mine and 200 liters of process water of lead and zinc flotation plant were prepared for flotation tests from the KZG (Kimia Zanjan Gostaran) plant. Process water sampling was performed in three stages and three different weeks from a flotation return water tanker, which was a combination of water thickeners, filter presses, and tailings dam water. It is noticeable that this water returns to the process water cycle

after settling in the stabilization ponds. For Galena flotation, zinc sulfate with 30% purity, Potassium Ethyl Xanthate (PEX) with 90% purity of Merck Company, and Methyl Isobutyl Carbinol (MIBC) were used as the depressant, Collector and Frother, respectively. In Sphalerite flotation, lime with 90% purity, copper sulfate 25%, Potassium Amyl Xanthate (PAX) with 90% purity, made by Merck and MIBC were used as pH modifier, activators, Collector and frother, respectively. Tap water was used for comparing the results of flotation experiments with process water.

2-2- Methods and Equipment

2-2-1- Sample preparation

The primary crushing was done by a jaw crusher made in China with an electric motor power of 7.5 kW and a CSS of 30 mm. The jaw crusher's product was fed the Cone crusher made in the Czech Republic with a power of 2.5 kW. The crushing section's final product was milling by a ball mill made in the USA with a power of 3 kW to the particle size of $d_{80}=150$ micron. This sample was divided into small samples using riffles and used for flotation experiments. Part of this sample was separated and used for identification analysis.

2-2-2- Identification Studies

500 ml of process water and Tap water sample were prepared for ICP-MS analysis. Environmental analysis of Suspended Solids, Chemical pollution, and Biological pollution (BOD, COD, and TSS) was performed on process water. A certain amount of sulfide ore sample after grinding was prepared and sent for XRF, XRD analysis, ore chlorine, PbO/ZnO analysis.

3- THE COMPARATIVE EFFECT OF PROCESS WATER WITH TAP WATER ON GALENA AND SPHALERITE FLOTATION

To perform these experiments, 400 grams of ore sample were added into a 1.5-liter flotation cell, and the preparation process was performed for 7 minutes. After adjusting the activating chemicals, pH, collector, and depressant were added to the pulp after a specific preparation time. Then the Frother was added to the pulp with a specific preparation time, finally, by opening the air valve,

getting the froth at a particular time performed. The sections of froth and the residual of the cells dried and weighted. Eventually, sent to the laboratory for chemical analysis. The quantities of chemicals and the pH preparation and adjustment steps were similar to those used in the KZG plant's industrial scale.

3-1- Comparative experiments of Galena flotation in the presence of Tap water and process water

Galena flotation experiments were performed with a constant pH of 8 and pulp preparation time and chemical residence time of 7 and 2 minutes. The conditions for Galena flotation tests were reported in Table 1.

In these experiments, to better compare the flotation process in the presence of process water and Tap water, experiments were performed with different conditions, and in each experiment, one parameter was changed. All conditions of each comparative experiment of two types of water were performed the same. In the Galena flotation experiments, the effect of changes in concentration of zinc sulfate as a depressant, potassium ethyl xanthate as a collector, frother, and the temperature of tests was investigated by using two types of water. After performing the initial flotation test and the possibility of more froth removing and increasing the recovery, from the second experiment, the froth releasing time was increased from 3 to 5 minutes, and this time was constant until the end of the experiments. In the second experiment, the concentration of

Table 1. Conditions of Galena rougher flotation tests

Parameters / unit	Number of Experiments					
	1	2	3	4	5	6
Zinc Sulfate (g/t)	1700	1700	2400	2400	4500	2400
PEX (g/t)	30	50	50	50	50	50
MIBC (g/t)	30	30	30	50	50	50
Experiments Temperature (°C)	23	23	23	23	23	35
Froth loading Time (min)	3	5	5	5	5	5

potassium ethyl xanthate as the collector was increased to 50 g/ton. The third experiment amount of zinc sulfate reached 2.4 kg/ton. In the fifth experiment, the amount of zinc sulfate reached 4.5 kg/ton (due to reducing the amount of zinc in lead concentration and reducing non-selective flotation). Since in the fifth experiment, the grade of Sphalerite reduced (which is the next stage of differential flotation) in the sixth experiment again the amount of 2400 g/ton was used. The fourth experiment was performed and evaluated by increasing the Frother to 50 g/ton. The sixth experiment was performed at 35 °C. The purpose was to experiment by changing the temperature parameter. Seasonal temperature changes for the Zanjan flotation plant are between -15 to 35 °C (according to meteorological reports of Zanjan province) since it was not possible to prepare the laboratory environment at low temperatures, the tests were done at 35 °C). At the time of froth removing and descending the cell froth level, the certain required water was added to the cell.

3-2- Comparative experiments of Sphalerite flotation in the presence of Tap water and process water

These experiments were performed with constant pH and residence time values of 9.5 and 2 minutes, respectively. As in the Galena stage in these experiments, to better compare, experiments were performed with different conditions and in each experiment, one parameter was changed. The conditions of the Sphalerite flotation tests are reported in Table 2. In these experiments, the effect of changes in copper sulfate and potassium amyl xanthate concentration and the experiment's temperature was investigated by using two types of water. Same as the Galena flotation test, in this section, froth removing of the cell was performed in 5 minutes from the second experiment because of the more froth loading.

4- RESULTS AND DISCUSSION

4-1- Analyze results of the process water and Tap water

According to the following tables, the results of identification tests for process water and Tap water are shown. The standard values of biological, chemical, and suspended solids parameters of water to release into nature are 100, 200, and 50

Table 2. Conditions of Sphalerite rougher flotation tests

Parameters / unit	Number of Experiments					
	1	2	3	4	5	6
Copper Sulfate (g/t)	2500	2000	3000	3000	3500	3000
PAX (g/t)	150	150	150	200	200	200
Experiments Temperature (°C)	23	23	23	23	23	35
Froth loading Time (min)	3	5	5	5	5	5

mg/l, respectively, which in the process water all of these parameters were illegal [21]. Most of the pollution of process water was related to chemical pollution due to the use of flotation reagents. Due to the high pollution of the process water, although 5-15% of Tap water is added in every cycle, processing plants need to drain the process water and charge more Tap water after several cycles. In Table 3 and Figure 1, illegal amounts of processed water from an environmental perspective are shown. In Table 4, the results of chemical analysis of Tap water and process water by the ICP-MS method are shown. The results can represent the extent of process water pollution. Ionic pollution of water leads to flotation disruption, by unwanted reactions with minerals, sedimentation of illegal ions on the valuable mineral particles and lack of access to the surface of the valuable particles, reaction with gangue and making them

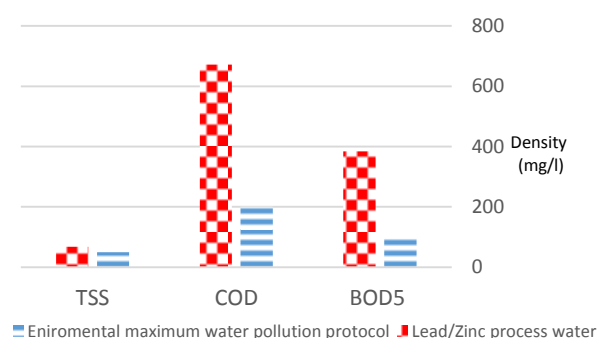


Figure 1. Comparison of the maximum limits of pollution, according to the environmental instructions with the amount of flotation process water [10]

Table 3. Environmental chemical analysis of process water and standard limits *

Row	Environmental Experiments			
	Name	BOD ₅ (mg/lit)	COD (mg/lit)	TSS (mg/lit)
1	Environment maximum water pollution protocol	BOD ₅ <100	COD<200	TSS<50
2	Process Water of flotation	384	672	68

* According to provision 5 of the By-Laws on Prevention of Water Pollution and according to provision 3 of the same By-Laws and with the cooperation of the Ministries of Health, Treatment and Medical Education, Energy, Industries and Mines and Metals and Agriculture by the Environmental Protection Organization Biology has been compiled and prepared [10]

Table 4. ICP-MS analysis of the tap water and process water

Element/Parameter	Amount in the Tap water (mg/l)	Amount in the process water (mg/l)
Calcium (Ca)	42.5	352
Magnesium (Mg)	7.90	96.50
Lead (Pb)	0.01	1.07
Iron (Fe)	0.3	1.75
Copper (Cu)	0.03	0.21
Zinc (Zn)	2.20	18.27
Chlorine (Cl)	20	1004
Potassium (K)	133	358
Sodium (Na)	192	5431
Sulfur (S)	167	5588
pH	6.8	7.9

hydrophobic, leading to the formation of metal and non-metallic complexes and salts and also increase the viscosity of the reaction medium. In Table 4, the most ionic pollution of process water is related to sulfur, sodium, chlorine, and calcium. The amount of elements in process water and ionic pollution is due to the addition of ore elements and ions to the water and chemical reagents during the flotation process. Table 5 reports the origin and formation cause of ions high pollution in process water which depends on the chemical reagents consumption and ore elements.

4-2- Results of identifying Galena and Sphalerite samples

The mineralogical study of the sulfide ore sample represents that the significant minerals are smithsonite and quartz. The minor minerals are Sphalerite, Hemimorphite, Calcite, Goethite, Galena, Cerussite, Kaolinite, and Muscovite. Table 6 reports the XRF analyze and the amount of

Galena and Sphalerite, according to Table 7, were reported as 1.74 and 7.06 percent, respectively.

4-3- Results of Galena and Sphalerite flotation in the presence of Tap water and process water

According to Figures 2 and 3, the low quality of process water decreases the flotation process's grade and recovery compared to Tap water. Also, flotation with process water was with high and different dimensions froth, which can disrupt the flotation process. Galena grade was 9% lower than flotation with Tap water, and its recovery was 7% lower. In the flotation of Sphalerite, the grade increased by 2% compared to Tap water, and its recovery decreased by 5%. This decrease in the grade of Sphalerite compared to process water could be due to the froth removal time, which was increased from 3 to 5 minutes from the second experiment. According to previous research, the high froth and bubbles of different sizes are probably due to the presence of ions and residual

Table 5. Illegal amount of elements in process water and their amount in mine ore and flotation chemicals

Element	Element value in the process water (ppm)	Amount in the mine ore %	Element value in the flotation agents and their consumption in the plant
Ca	352	3.6	71% from CaO, average consumption 1500 g/t
Na	5431	0.15	42% from Na ₂ S, average consumption 4000g/t
K	315	0.78	19% from PAX and 23.5% from PEX, Average consumption of PAX 250 g/t and PEX 20 g/t
S	5588	6.83	58% from Na ₂ S, average consumption 4000g/t
Cl	1004	0.06	-

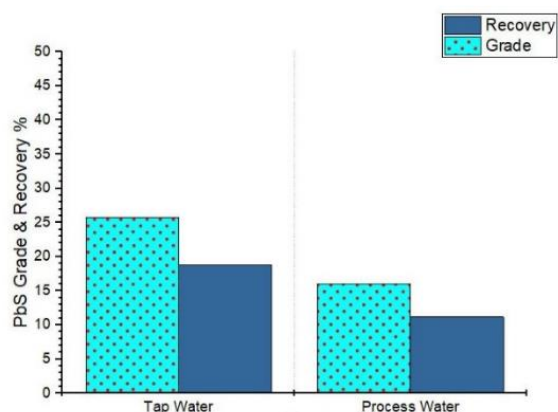
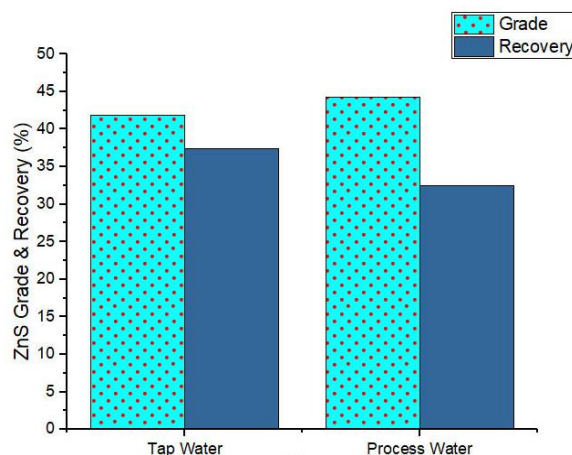
Table 6. XRF analysis of Anguran's mine ore with PbS/PbO and ZnS/ZnO Analyze

Elements/Oxides	ZnO	ZnS	SiO ₂	Fe ₂ O ₃	PbO	PbS	CaO
%	20.75	7.06	23.71	5.37	1.05	1.74	3.60

Table 7. XRD analysis of Anguran's mine ore

Major phase(s)			
Smithsonite			
Quartz			
Minor phase(s)			
Hemimorphite	Goethite	Galena	Muscovite - illite
Sphalerite	Calcite	Cerussite	Kaolinite

complexes of soluble reactants (such as sulfur, sulfate composition, and water-soluble salts) [7]. Comparative Figures of the grade and recovery results of the Galena and Sphalerite flotation experiments can be seen in Figures 4 to 13. In the second experiment, the ZnS grade decreased significantly compared to the first test experiment, which could be due to the decrease in the copper sulfate as the activator and increasing the froth removal time, which increased the non-selective flotation. With the same conditions, the grade of ZnS has increased, and this indicates that it is possible to float with fewer chemicals compared to process water and save on chemical consumption. The reason for the increase in the use of chemicals agents in flotation with process water can be the interaction of illegal ions in water with minerals and flotation reactants and competition with chemicals agents for adsorption on the surface of valuable minerals.

**Figure 2. Comparison of grade and recovery in Galena flotation with tap and process water in the first experiment****Figure 3. Comparison of grade and recovery in Sphalerite flotation with tap and process water in the first experiment**

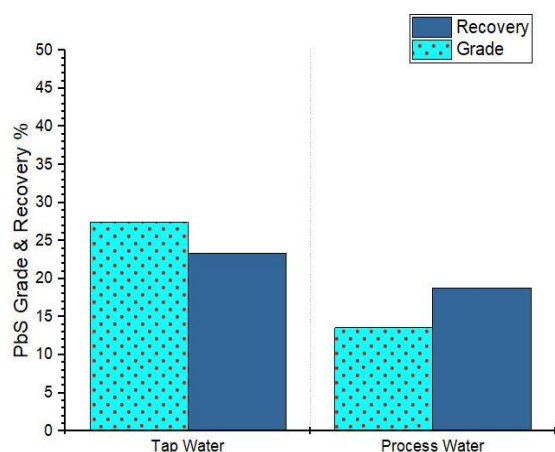


Figure 4. Comparison of grade and recovery in Galena flotation with tap and process water in the second experiment

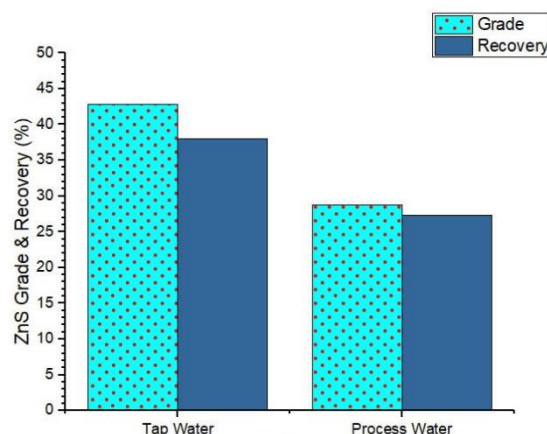


Figure 7. Comparison of the grade and recovery of Sphalerite flotation with tap water and process water in the third experiment

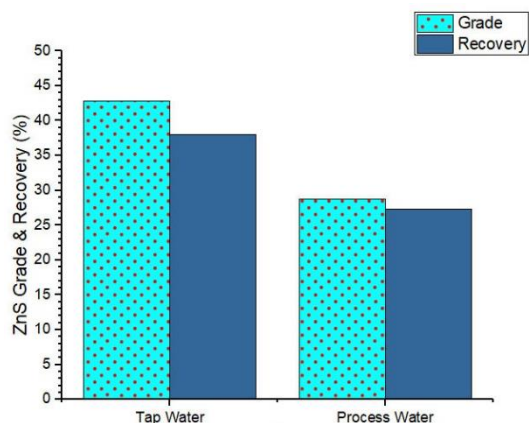


Figure 5. Comparison of grade and recovery in Sphalerite flotation with tap and process water in the second experiment

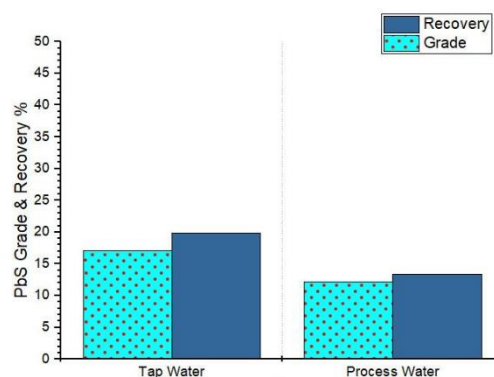


Figure 8. Comparison of grade and recovery in Galena flotation with tap water and process water in the fourth experiment

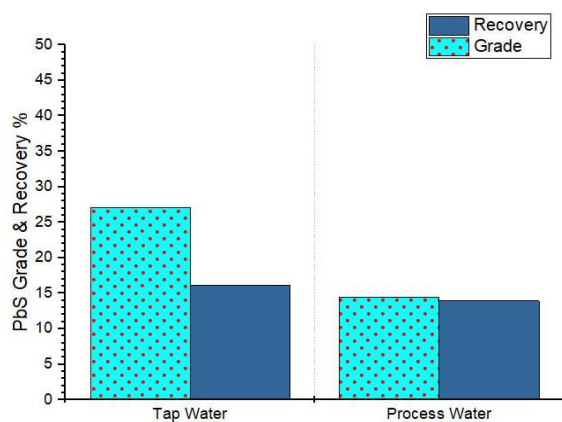


Figure 6. Comparison of grade and recovery of Galena flotation with tap water and process water in the third experiment

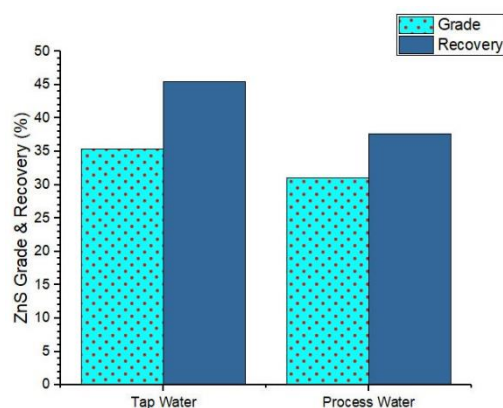


Figure 9. Comparison of grade and recovery in Sphalerite flotation with tap water and process water in the fourth experiment

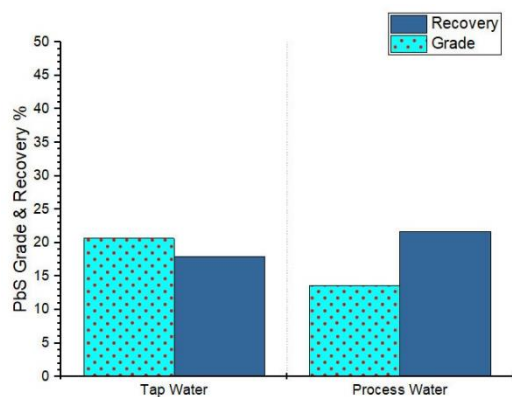


Figure 10. Comparison of grade and recovery in Galena flotation with tap water and process water in the fifth experiment

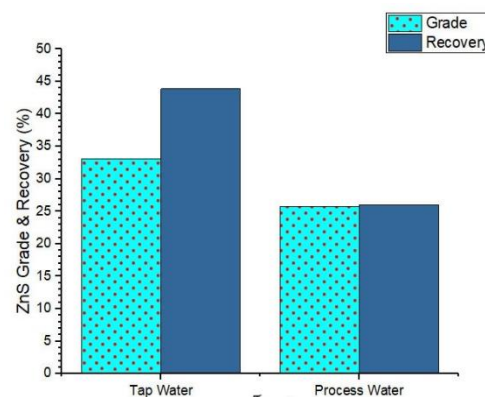


Figure 13. Comparison of grade and recovery in Sphalerite flotation with tap water and process water in the sixth experiment

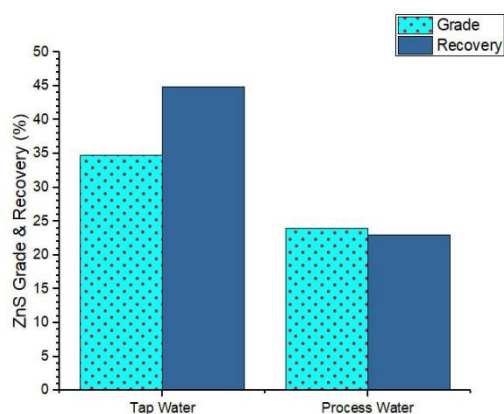


Figure 11. Comparison of grade and recovery in Sphalerite flotation with tap water and process water in the fifth experiment

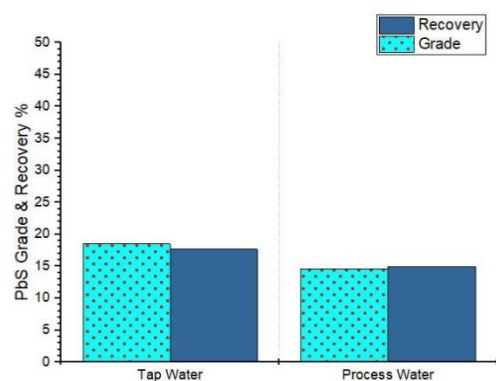


Figure 12. Comparison of grade and recovery in Galena flotation with tap water and process water in the sixth experiment

It is observed that due to more use of zinc sulfate depressant to achieve higher lead grade and decrease the zinc grade in lead concentrate, the amount of lead recovery has decreased, which can be due to increasing the sulfate ion in water and formation of lead sulfate which is a hydrophilic compound. With the increase of sulfate ions and the formation of lead sulfate, lead was inactivated, and its recovery was reduced. In Sphalerite flotation, the difference in grade and recovery of the flotation process with Tap water compared to process water is quite clear and indicates the low quality of process water for sulfide flotation. A noticeable point in the results of concentrate analysis is the high amount of non-selective elements in concentrates with process water, as shown in Table 8. According to XRF analysis, the number of various oxide elements mentioned, which has been floated and in the concentrate with process water, is more than the same oxides in the concentrate with Tap water flotation process. While the amount of sulfur, unlike other values, is lower in the concentrate with process water (in Galena and Sphalerite, 13.58% and 14.77%, respectively), it is due to a lower grade in concentrates with process water. Due to the sulfur present in Galena's chemical formula (PbS) and Sphalerite (ZnS), which always, by increasing the grade of the concentrate, the amount of sulfur increases proportionally. Oxidation and reduction reactions and making oxide minerals hydrophobe is due to reaction with free sulfur particle during flotation

Table 8. Amounts of different oxides in floating sections with tap water and process water

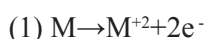
Water Type	Concentrate	SiO ₂ (%)	CaO (%)	K ₂ O (%)	MgO (%)	MnO (%)	LOI (%)	TiO ₂ (%)	S (%)
Process Water	PbS	11.42	1.69	0.82	0.65	0.06	16.99	0.14	13.58
	ZnS	12.30	1.80	0.91	0.64	0.06	17.70	0.15	14.77
Tap Water	PbS	5.38	0.76	0.21	0.3	<0.05	8.41	0.08	20.54
	ZnS	4.13	0.47	0.15	0.21	<0.05	14.28	0.05	27.63

Table 9. The results of the fourth to sixth rougher flotation tests

Tap Water	Fourth Stage	Investigation	PbS	ZnS
		PbS Grade	17.08	6.08
		Recovery	19.88	45.45
		Zinc Grade	19.25	35.38
	Fifth Stage	PbS Grade	20.63	5.94
		Recovery	17.90	44.91
		Zinc Grade	13.28	34.75
	Sixth Stage	PbS Grade	18.48	8.47
		Recovery	17.64	43.85
		Zinc Grade	10.40	33.07
Process Water	Fourth Stage	PbS Grade	12.2	5.15
		Recovery	13.38	37.63
		Zinc Grade	19.3	31
	Fifth Stage	PbS Grade	13.61	8.76
		Recovery	21.68	22.93
		Zinc Grade	15.03	23.94
	Sixth Stage	PbS Grade	14.60	8.01
		Recovery	14.97	26.01
		Zinc Grade	12.60	25.69

in the presence of process water shown in the following chemical formulas. The sulfur of sulfide minerals, release after oxidation in the pulp, and its electrons reduced cationic minerals such as lead and zinc and compete with flotation agents for adsorption on the surface of lead, zinc. Sulfur particles in reaction with other oxides cause their activation and hydrophobicity, which leads to low grade and recovery.

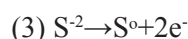
Minerals Oxidation



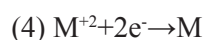
Sulfide Minerals Oxidation



Anions Oxidation



The reaction of Cationic Minerals with Released Anions and reduction of minerals



Where M represents Pb, Zn, ... and MS represents PbS, ZnS, ...

In Table 9, the results of the fourth to sixth rougher flotation tests are shown. Process water produces salt and collector complexes in the pulp, increasing the stability of the froth and viscosity. The undesirable amount of calcium and

sodium ions has a negative effect on the stability of the froth phase and viscosity. In the presence of various metal ions, the stability of the frother increases [7]. Also, probably excess metal cations precipitate as colloidal particles that may act as a reservoir to the activation of metal cations in process water [11].

The Results of Galena and Sphalerite flotation rougher experiments with process water and tap water are reported in Table 10. In the first experiment, as previously mentioned, at the froth loading time of the rougher flotation with process water, the Sphalerite grade was slightly higher than the sulfide grade with Tap water in 3 minutes. After increasing the time to 5 minutes, it was determined that it is possible to increase the zinc grade in the flotation with Tap water by increasing the condition time. It seems that with the increase in PAX consumption in the fourth experiment, the recovery of Sphalerite flotation in process water has an upward trend and indicates a higher consumption of chemicals in process water to closing the results of Tap water. In the fifth experiment, the Galena recovery in Tap water was less than with process water, which could be due to more zinc sulfate to prevent non-selective zinc flotation in the Galena flotation stage. The high amount of sulfate ions in Tap water decreases the recovery of lead, but in process water, it has been increasing the Galena recovery, which indicates getting closer to the results of Tap water needs more agent consumption when process water was used.

5- CONCLUSION

In this paper, the effect of lead and zinc process water on the grade and recovery of Galena and Sphalerite flotation was investigated and compared with Tap water. After identification studies of process water samples, it was found that this water has high ion pollution and illegal environmental pollution. Galena flotation tests showed that the grade and recovery in using Tap water were 8% higher and 3% higher than the process water, respectively. Also, the flotation results on Sphalerite showed higher grade and recovery of zinc by 10 and 11%, respectively, compared to process water, which is a considerable amount. It is suggested that comparative experiments be performed on a pilot plant scale to compare flotation results with Tap water and process water

Table 10. Results of Galena and Sphalerite flotation rougher experiments with process water and tap water

Flotation Type	Investigation	Process Water	Tap Water
PbS	Recovery	15.66	18.94
	Pb Grade	14.09	22.75
	Zn Grade	20.16	21.36
ZnS	Recovery	29.23	40.64
	Pb Grade	30.23	40.65
	Zn Grade	7.18	7.33

of lead and zinc flotation more accurately.

After performing comparative experiments on a pilot plant scale and the same result of laboratory scale, it is recommended to treat the process water and use it again in the processing process.

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