

Recovery of Hydrogen Sulfide, Copper and Cobalt from the Samples after Conversion of Pyrite to FeS

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The aim of this study was to recover hydrogen sulfide, copper and cobalt from the pyrite samples provided from Ergani and Küre Copper Mining Co., Turkey. The samples were subjected to two different processes: in first process, the sample was mixed with different rates of iron at varying temperature and time and then subjected to roasting process in closed vessel system. In the second process, the sample was directly exposed to roasting process in a closed vessel system. In both processes, conversion values of pyrite into FeS were determined. After direct roasting the pyrite at 725°C for 1 h, the conversion rate of pyrite into FeS was found to be 98.9 ± 0.1 %. By addition of H₂SO₄ the sample converted into FeS structure, H₂S gas was formed. Through reaction of water with the residue remaining after separation of H₂S gas from solid phase, iron passed into solution, while copper and cobalt remained in the solid phase. In order to recover cobalt and copper from the sample in which iron was removed, two procedures were followed; first procedure includes taking cobalt and copper in solution by direct roasting the sample in air atmosphere at 600°C. The second process involves taking cobalt and copper in solution medium through roasting the sample under the same conditions after enrichment with flotation. As a result of roasting the samples enriched with flotation, the optimal conditions in which copper and cobalt could be taken into solution with high yield were determined.

Key Words: Copper, Cobalt, Hydrogen sulfide, Pyrite, Roasting.

INTRODUCTION

Copper oxide and sulphide ores mainly are known as tenorite malachite, azurite, enargite, chalcopyrite, chalcocite, covellite, bornite, brochantite. Among the copper sulphide minerals, chalcocite (Cu₂S), pyrite (FeS₂) are the easiest dissolving minerals and chalcopyrite (CuFeS₂) is the most abundant, the most refractory, and the most difficult leaching mineral¹.

Leaching of copper sulphides like chalcopyrite at strong and weak acidic medium has been investigated²⁻⁷. The work focused on chalcopyrite concentrated and its slag with dissolution by various leachants including sulfuric acid, ammonia, hydrochloric acid, acetic acid and cyanide. Atmospheric and high-pressure leaching was also investigated⁸⁻¹².

Some of the processes developed for copper can also be extended to treatment of complex sulphides. One of such processes is Sherritt-Cominco copper process. It was developed to treat copper sulphide concentrates through a moderate pressure autoclave leaching. In this process, the important objective was to recover sulphur in its elemental form and not as sulphur dioxide or sulphate in order to ensure improved hygienic conditions and a negligible impact on the environment. A high-grade copper sulphide concentrate is produced after the removal of iron, which contains all of the initial precious metals. This concentrate is then leached under oxygen pressure to produce elemental sulphur and CuSO_4 solution¹³.

The pressure leaching has been commercially used in the metal ores and concentrates (copper, zinc, gold, alumina, nickel, uranium, molybdenum, cobalt, tungsten and tin)¹⁴⁻¹⁷. In other study, under optimum conditions, more than 95% of copper could be recovered by only a limited recovery of nickel, cobalt and zinc could be achieved by roasting the pre roasted slag with pyrite at 550°C for 1 h with a 0.25 pyrite/slag ratio. Around 2 % of the iron in the slag was extracted into the leach solutions¹⁸.

In the first step, FeS was produced from pyrite. After the addition of H_2SO_4 , FeS produced in the first step. From this reaction, H_2S was produced and only iron passed into solution. In addition, FeSO_4 was removed from solid residue by washing with water. At the final step, copper and cobalt was taken to the aqueous medium by roasting of solid residue.

The aim of hydrogen sulfide production is to realize formation of suitable surface occurred by sulphurisation procedure and method developed in our laboratory before flotation of variable copper ores. Pyrite is a waste produced with flotation of copper ores. Furthermore, the cost of hydrogen sulfide production can be decreased by using this method¹⁹⁻²².

EXPERIMENTAL

Experiments were performed on pyrite samples assured from Ergani situated in the South Eastern of Turkey and Küre situated in the Western Black Sea Region of Turkey. The elemental contents of the pyrite samples obtained from Ergani and Küre Copper Corporations is given that Table-1.

In the Ergani and Küre deposits, the main ore minerals are pyrite and chalcopyrite. In addition to that there are quartz, gold, carbonate, clay minerals like gangue and some important rare minerals².

TABLE-1
ANALYSIS OF SULFUR, COPPER, COBALT FOR PYRITE SAMPLES

Ores	Sulfur (%)	Copper (%)	Cobalt (%)
Ergani	43.30	0.66	0.25
Küre	41.40	0.50	0.12

The experiments were carried in a laboratory equipped with facilities for carrying out hydrometallurgical operations such as roasting and autoclave leaching process. The composition of all solutions and samples were determined from the laboratory experiments using atomic absorption spectrophotometer (Unicam 929) and sulphur was estimated by gravimetric Carlo-Erba 1108 model elemental analysis instrument. In the roasting experiments, a laboratory tunnel furnace (Carbolite) was employed. Autoclave experiments were done in a 1.3 L corrosion resistant reactor, which could be heated up to a maximum of 300°C. All the chemicals used were analytical grade (Merck) and all solutions were prepared with deionizer water.

The samples were grinded to -100 mesh and dried at 110°C. For the samples taken from Küre, first level of experiment was carried out in the steel reactor with 50 g capacity. Experimental processes were performed by heating the steel reactor in furnace to control the temperature. For pyrite samples taken from Ergani Copper Mining Corporation, experiments were carried out in the reactor with 8 kg capacity under nitrogen atmosphere and in a tunnel furnace.

RESULTS AND DISCUSSION

Conversion of FeS₂ to FeS

Hydrogen sulfide can neither be produced by directly addition of acid nor reducing with acid addition into solution medium. The production of hydrogen sulfide was investigated by converting of pyretic sulfur to sulfide sulfur. For this purpose two following ways were performed. The first way was done using reaction:



In these experiments, pyrite/iron were mixed different ratios at various temperatures and results were given in Table-2. As seen in Table-2, maximum conversion value was obtained by mixture ratio of 1.5/1 at 700°C. In the second way, studies were performed according to reaction below²⁰.



Pyrite was first treated to roasting process in the medium without air. Since maximum conversion of this process was obtained at 700°C, two different experiments at 700 and 725°C were to determine the conversion values from pyretic sulfur to sulfide sulfur. It was determined that 93.8 ± 0.5 % of pyretic sulfur was converted to sulfide sulfur at 700°C and 98.9 ± 0.1 % of pyretic sulfur was converted to sulfide sulfur at 725°C. The sulfur separated from the medium as the result of this experiment was collected in a different container and showed that the reaction equation (2) occurred. It is possible to conclude that by roasting pyrite in absence of air at 725°C for 1 h, appropriate results can be obtained. Converting value was calculated as the ratio of sulfide sulfur obtained from pyrite and total sulfur.

TABLE-2
SULPHIDE VALUES BY ROASTING OF PYRITE/IRON MIXTURE AT
VARIOUS TEMPERATURES

Mixture ratio (pyrite/iron)	Temp. (°C)	Transformation (%)	Mixture ratio (pyrite/iron)	Temp. (°C)	Transformation (%)
1.5/1	500	34.20 ± 0.11	2/1	500	30.64 ± 0.38
	525	39.82 ± 0.18		525	33.75 ± 0.22
	550	45.46 ± 0.28		550	41.11 ± 0.18
	575	53.64 ± 0.24		575	49.63 ± 0.46
	600	61.19 ± 0.21		600	57.69 ± 0.41
	625	73.68 ± 0.34		625	64.82 ± 0.25
	650	88.73 ± 0.19		650	82.26 ± 0.29
	675	95.20 ± 0.25		675	91.43 ± 0.69
	700	99.73 ± 0.36		700	95.21 ± 0.14
2.5/1	500	27.54 ± 0.35	3/1	500	19.37 ± 0.78
	525	29.30 ± 0.29		525	21.15 ± 0.54
	550	37.43 ± 0.48		550	26.70 ± 0.68
	575	42.64 ± 0.16		575	36.54 ± 0.22
	600	54.71 ± 0.28		600	43.12 ± 0.32
	625	57.23 ± 0.62		625	48.54 ± 0.18
	650	67.47 ± 0.35		650	51.37 ± 0.27
	675	79.43 ± 0.39		675	61.40 ± 0.54
	700	83.58 ± 0.21		700	76.82 ± 0.23
3.5/1	500	17.44 ± 0.12	4/1	500	13.50 ± 0.65
	525	18.09 ± 0.54		525	14.49 ± 0.72
	550	24.23 ± 0.63		550	19.41 ± 0.47
	575	30.60 ± 0.21		575	26.65 ± 0.85
	600	39.83 ± 0.42		600	35.05 ± 0.26
	625	41.54 ± 0.58		625	37.69 ± 0.63
	650	47.64 ± 0.09		650	42.44 ± 0.21
	675	54.98 ± 0.25		675	46.19 ± 0.39
	700	61.60 ± 0.62		700	51.02 ± 0.45

Recovery of copper and cobalt from treated and raw pyrite

It is clear from the second method that almost all of the impurities release from the pyrite and then pure H₂SO₄ can be produced from SO₂, which is extracted from treated pyrite roasting in presence of air at 600°C. By reacting residue with water, copper and cobalt can be passed to solution²¹. In order to compare raw pyrite obtained from Ergani Copper Corporation to treated pyrite, raw pyrite was roasted with air at 600°C, then amount of copper and cobalt passed to the aqueous solution was determined and the results were given in Table-3.

The maximum ratios of copper and cobalt passed to the solution were 51.4 ± 0.2 and 29.2 ± 0.2 %, respectively. Since these results were insufficient, values of copper and cobalt passed to the solution by roasting the

treated pyrite at 600°C at different duration was examined and the results were given in Table-4.

TABLE-3
THE VALUES OF COPPER AND COBALT TAKEN INTO THE AQUEOUS SOLUTION BY ROASTING OF RAW PYRITE AT 600°C

Roasting Time (h)	Amount taken into solution and the residue					
	Copper (%)			Cobalt (%)		
	a	b	C	a	b	c
1	4.6 ± 0.7	-	95.4 ± 0.5	2.9 ± 0.7	-	97.1 ± 0.3
2	9.1 ± 0.5	-	90.9 ± 0.1	8.8 ± 0.1	-	91.2 ± 0.4
3	19.9 ± 0.3	-	80.1 ± 0.2	15.2 ± 0.3	-	84.8 ± 0.6
4	28.7 ± 0.4	-	71.3 ± 0.5	20.1 ± 0.2	-	79.9 ± 0.8
5	39.2 ± 0.4	-	60.8 ± 0.3	24.4 ± 0.5	-	75.6 ± 0.8
6	51.4 ± 0.2	-	48.6 ± 0.4	29.2 ± 0.3	-	70.8 ± 0.7
7	50.9 ± 0.2	-	49.1 ± 0.2	30.1 ± 0.1	-	69.9 ± 0.7

- a. Yield of leaching by water (%).
b. Yield of leaching by 0.1 M H₂SO₄ (%).
c. The amount of unleached.

Table-4 shows that the maximum values were obtained from the samples roasted for 6 h. It was observed that 82.2 ± 0.5% of Cu was extracted into aqueous solution, 10.9 ± 0.5% in 0.1 M H₂SO₄ and 6.9 ± 0.2% of Cu was remained, 88.2 ± 0.3% of Co was extracted into aqueous solution, 6.7 ± 0.5% in 0.1 M H₂SO₄ and 5.1 ± 0.5% of Co was remained. It was concluded that after the roasting of pyrite treated at 600°C, ca. 5-6 % of Cu and Co remained in residue.

TABLE-4
THE VALUES OF COPPER AND COBALT TAKEN INTO THE SOLUTION BY ROASTING OF TREATED PYRITE SAMPLE AT 600°C

Roasting Time (h)	Amount taken into solution and the residue					
	Copper (%)			Cobalt (%)		
	a	b	c	a	b	c
1	18.5 ± 0.8	40.8 ± 0.5	40.7 ± 0.1	59.5 ± 0.7	3.4 ± 0.7	37.1 ± 0.4
2	48.5 ± 0.2	26.8 ± 0.1	24.7 ± 0.2	73.7 ± 0.6	13.1 ± 0.1	13.2 ± 0.5
3	72.1 ± 0.3	21.0 ± 0.7	6.8 ± 0.9	83.6 ± 0.4	14.3 ± 0.4	2.1 ± 0.3
4	78.1 ± 0.4	20.1 ± 0.4	1.8 ± 0.2	94.3 ± 0.9	3.9 ± 0.1	1.8 ± 0.4
5	80.2 ± 0.2	15.1 ± 0.2	4.7 ± 0.3	93.4 ± 0.1	4.3 ± 0.8	2.3 ± 0.2
6	82.2 ± 0.5	10.9 ± 0.5	6.9 ± 0.2	88.2 ± 0.3	6.7 ± 0.5	5.1 ± 0.5
7	82.4 ± 0.1	7.6 ± 0.9	10.0 ± 0.1	83.7 ± 0.6	11.3 ± 0.4	5.0 ± 0.1

Recovery of copper and cobalt from the residue after production of hydrogen sulfide

The residue obtained after production of hydrogen sulfide was washed by pure water and ferrous sulfate was taken into aqueous solution. After that, residue without ferrous sulfate containing 2 % Cu and 0.56% Co was roasted at 600°C at different duration and results were given in Table-5.

TABLE-5
THE VALUES OF COPPER AND COBALT TAKEN INTO THE SOLUTION BY ROASTING AT 600°C AFTER THE PRODUCTION OF HYDROGEN SULFIDE

Roasting Time (h)	Amount taken into solution and the residue					
	Copper (%)			Cobalt (%)		
	a	b	c	a	B	c
1	6.5 ± 0.3	23.4 ± 0.1	70.1 ± 0.1	11.2 ± 0.9	43.2 ± 0.5	45.6 ± 0.2
2	17.6 ± 0.2	21.3 ± 0.9	61.1 ± 0.5	22.9 ± 0.7	40.4 ± 0.2	36.1 ± 0.6
3	33.0 ± 0.1	14.2 ± 0.3	52.8 ± 0.2	24.2 ± 0.1	40.1 ± 0.2	35.7 ± 0.5
4	56.8 ± 0.2	11.2 ± 0.8	32.0 ± 0.4	34.2 ± 0.2	37.1 ± 0.6	28.7 ± 0.8
5	70.7 ± 0.1	10.4 ± 0.7	18.9 ± 0.2	38.5 ± 0.7	36.2 ± 0.9	25.3 ± 0.1
6	76.2 ± 0.2	9.2 ± 0.1	14.6 ± 0.1	46.3 ± 0.4	33.3 ± 0.1	20.3 ± 0.3
7	74.1 ± 0.2	8.6 ± 0.1	17.3 ± 0.2	48.2 ± 0.7	32.7 ± 0.5	19.1 ± 0.4

It was observed that the extraction of copper and cobalt in this way did not result in high yield. As well as samples without removing ferrous sulfate after hydrogen sulfide production were roasted at 600°C and the results are given in Table-6.

TABLE-6
THE VALUES OF COPPER AND COBALT TAKEN INTO THE SOLUTION BY ROASTING THE SAMPLE THAT FERROUS SULFATE WAS NOT REMOVED

Roasting Time (h)	Copper (%)			Cobalt (%)		
	a	b	c	a	b	c
5	68.9 ± 0.2	-	31.1 ± 0.7	79.9 ± 0.3	-	20.1 ± 0.2
6	72.1 ± 0.4	-	27.9 ± 0.3	80.1 ± 0.6	-	19.9 ± 0.1
7	73.2 ± 0.5	-	26.8 ± 0.1	85.9 ± 0.5	-	14.1 ± 0.4

It is clear that extraction of cobalt and copper did not result in high yield; hence, sample (FeSO₄ removed) mixed with FeSO₄ and treated pyrite with different ratios was roasted and results were given in Tables 7 and 8.

TABLE-7
THE VALUES OF COPPER AND COBALT TAKEN INTO THE SOLUTION BY ROASTING AT 600°C CONSIST OF VARIOUS AMOUNTS OF FERROUS SULFATE AND TREATED PYRITE TO 10 g OF WASTE (ROASTING TIME 6 h)

Added Amount of FeSO ₄ (g)	Amount taken into solution and the residue					
	Copper (%)			Cobalt (%)		
	a	b	c	a	b	c
0.15	92.1 ± 0.3	1.2 ± 0.1	6.7 ± 0.2	73.3 ± 0.1	1.6 ± 0.1	25.1 ± 0.4
0.20	78.3 ± 0.1	4.9 ± 0.7	16.8 ± 0.5	83.9 ± 0.5	4.9 ± 0.4	11.2 ± 0.1
0.25	78.1 ± 0.5	7.7 ± 0.4	14.2 ± 0.5	84.2 ± 0.2	5.1 ± 0.1	10.7 ± 0.3
0.30	72.4 ± 0.3	10.3 ± 0.6	17.3 ± 0.1	85.8 ± 0.5	5.3 ± 0.6	8.9 ± 0.1
0.40	62.8 ± 0.5	12.3 ± 0.2	24.9 ± 0.6	86.8 ± 0.8	5.3 ± 0.4	7.9 ± 0.4
0.60	59.3 ± 0.3	15.2 ± 0.5	25.5 ± 0.2	86.5 ± 0.4	5.2 ± 0.5	8.3 ± 0.2

As seen in Tables 7 and 8, in order to obtain maximum yield for cobalt and copper, different amount of treated pyrite was added to the 10 g of sample, which was got after removing FeSO₄ obtained the production of

TABLE-8
COPPER AND COBALT VALUES TAKEN INTO THE SOLUTION BY ROASTING
AT 600°C CONSIST OF VARIOUS AMOUNTS OF TREATED PYRITE AND 10 g
WASTE (ROASTING TIME 6 h)

Added amount of treated pyrite (g)	Amount taken into solution and the residue					
	Copper (%)			Cobalt (%)		
	a	b	c	a	b	c
0.15	67.9 ± 0.3	10.2 ± 0.1	21.9 ± 0.2	86.4 ± 0.4	7.1 ± 0.1	6.5 ± 0.3
0.25	85.0 ± 0.1	8.2 ± 0.9	6.8 ± 0.4	89.8 ± 0.2	6.9 ± 0.8	3.3 ± 0.2
0.30	88.8 ± 0.1	5.9 ± 0.3	5.3 ± 0.2	88.6 ± 0.5	6.4 ± 0.3	5.0 ± 0.2
0.40	95.1 ± 0.4	1.6 ± 0.8	3.3 ± 0.5	91.1 ± 0.3	7.2 ± 0.7	1.7 ± 0.5
0.50	95.5 ± 0.5	1.7 ± 0.7	2.8 ± 0.6	91.4 ± 0.7	7.0 ± 0.5	1.6 ± 0.6
0.60	98.1 ± 0.4	1.5 ± 0.4	0.4 ± 0.2	92.3 ± 0.3	7.7 ± 0.3	–
0.70	97.6 ± 0.2	1.5 ± 0.1	0.9 ± 0.1	91.9 ± 0.2	8.0 ± 0.1	0.1 ± 0.3
0.80	96.1 ± 0.3	1.7 ± 0.5	2.2 ± 0.5	91.5 ± 0.4	7.9 ± 0.4	0.6 ± 0.4
0.90	95.3 ± 0.1	2.1 ± 0.6	2.6 ± 0.6	91.2 ± 0.3	8.1 ± 0.2	0.7 ± 0.7
1.00	94.9 ± 0.4	2.3 ± 0.3	2.8 ± 0.3	90.9 ± 0.5	8.4 ± 0.4	0.7 ± 0.2

hydrogen sulfide and this mixture was roasted at 6 h. It was determined that 100 ± 0.3 % of cobalt and 99.6 ± 0.4 % of copper were extracted into solution by adding 0.6 g of treated pyrite.

Recovery of copper and cobalt from concentrate with flotation of residue

The maximum rates of the copper and cobalt were taken into the solution, when the sample without ferrous sulfate was roasted with treated pyrite. Various experiments were made in order to observe different values of copper and cobalt taken to the solution. With the improved flotation method, the values of copper and cobalt were 5.19 and 1.09 %, respectively and the roasting process of this sample with treated pyrite at 600°C was made¹⁴. The results of this experiment were given at Table-9.

As seen from Table-9, 99.9 ± 0.3 % (a+b) of copper and 100 ± 0.4 % (a+b) of cobalt were extracted into solution by roasting the mixture of 10 g concentrated sample and 0.6 g treated pyrite at 6 h.

TABLE-9
THE VALUES OF COPPER AND COBALT TAKEN INTO THE SOLUTION BY ROASTING
THE MIXTURE OF SAMPLE ENRICHED BY FLOTATION AND TREATED PYRITE*

Elements (%)	Roasting time (h)								
	5			6			6		
	10 g Conc.			10 g Conc.			10 g Conc. + 0.6 g tp		
	a	b	c	a	b	c	a	b	c
Copper	80.6±0.2	14.6±0.2	4.8±0.4	84.5±0.6	11.8±0.1	3.7±0.2	97.6±0.3	2.3±0.3	0.1±0.3
Cobalt	91.8±0.4	6.1±0.3	2.1±0.3	92.0±0.4	7.6±0.2	0.4±0.1	93.1±0.4	6.9±0.4	–

tp: Treated Pyrite Conc: Concentrated sample

*Average for three independent experiments.

Conclusions

In this study, a new method was developed to obtain hydrogen sulfide, copper and cobalt from pyrite and this study was occurred at five stages. In

the first stage, sulfurization process was achieved by roasting the pyrite at optimum condition (at 725°C in airless medium for 1 h). In second stage, hydrogen sulfide was produced by adding stoichiometry amount of H₂SO₄ to the sample. In third stage, ferrous sulfate was removed from the sample obtained from stage two by washing with H₂O. In the fourth stage, the concentrate was obtained with the flotation of the sample got from stage three. In the last stage of this study, the mixture of a proper amount of the concentrate obtained from the stage four and treated pyrite (the mixture obtained from 10 g concentrate sample and 0.6 g treated pyrite) with the sample obtained by roasting at 600°C for 6 h was determined and then copper and cobalt were taken to the solution medium and 99.9 ± 0.3 % of copper and 100 ± 0.4 % of cobalt were extracted into solution.

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