ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ
Satbayev University

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
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NEWS

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN
Satbayev University

SERIES
OF GEOLOGY AND TECHNICAL SCIENCES

6 (450)

NOVEMBER – DECEMBER 2021

THE JOURNAL WAS FOUNDED IN 1940

PUBLISHED 6 TIMES A YEAR

ALMATY, NAS RK

Clarivate
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«ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы».
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Меншіктеуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» РҚБ (Алматы қ.).
Қазақстан Республикасының Ақпарат және қоғамдық даму министрлігінің Ақпарат комитетінде 29.07.2020 ж. берілген **№ KZ39VPY00025420** мерзімдік басылым тіркеуіне қойылу туралы куәлік.
Тақырыптық бағыты: геология, мұнай және газды өңдеудің химиялық технологиялары, мұнай химиясы, металдарды алу және олардың қосындыларының технологиясы.
Мерзімділігі: жылына 6 рет.
Тиражы: 300 дана.
Редакцияның мекен-жайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., тел.: 272-13-19
http://www.geolog-technical.kz/index.php/en/

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Типографияның мекен-жайы: «Аруна» ЖК, Алматы қ., Мұратбаев көш., 75.

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«Известия НАН РК. Серия геологии и технических наук».
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы).
Свидетельство о постановке на учет периодического печатного издания в Комитете информации Министерства информации и общественного развития Республики Казахстан **№ KZ39VPY00025420,** выданное 29.07.2020 г.
Тематическая направленность: геология, химические технологии переработки нефти и газа, нефтехимия, технологии извлечения металлов и их соеденений.
Периодичность: 6 раз в год.
Тираж: 300 экземпляров.
Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, оф. 219, тел.: 272-13-19
http://www.geolog-technical.kz/index.php/en/

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Адрес типографии: ИП «Аруна», г. Алматы, ул. Муратбаева, 75.

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News of the National Academy of Sciences of the Republic of Kazakhstan. Series of geology and technology sciences.
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Owner: RPA «National Academy of Sciences of the Republic of Kazakhstan» (Almaty).
The certificate of registration of a periodical printed publication in the Committee of information of the Ministry of Information and Social Development of the Republic of Kazakhstan **No. KZ39VPY00025420,** issued 29.07.2020.
Thematic scope: geology, chemical technologies for oil and gas processing, petrochemistry, technologies for extracting metals and their connections.
Periodicity: 6 times a year.
Circulation: 300 copies.
Editorial address: 28, Shevchenko str., of. 219, Almaty, 050010, tel. 272-13-19
http://www.geolog-technical.kz/index.php/en/

© National Academy of Sciences of the Republic of Kazakhstan, 2021

Address of printing house: ST «Aruna», 75, Muratbayev str, Almaty.

NEWS
OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES OF GEOLOGY AND TECHNICAL SCIENCES
ISSN 2224-5278
Volume 6, Number 450 (2021), 46-52 https://doi.org/10.32014/2021.2518-170X.118

UDC 66.022:53.097

Begalinov A., Shautenov M., Medeuov Ch., Almenov T., Bektur B.

Satbayev University, Almaty, Kazakhstan.
E-mail: bekturbek@bk.ru

MECHANOCHEMICAL ACTIVATION OFTHE PROCESSING OF GOLD-BEARING SULFIDE RAW MATERIALS

Abstract. The mechanochemical activation of the gold-bearingflotation concentrate with the addition of copper sulfate in the grinding process has been studied. It has been established that as a result of the mechanical activation, gold is partially leached due to sulfur content in the source raw material, which acts as a leaching agent. At the stage of grinding and extraction of gold into solution was 48%.

Mechanical processing is one of the most important operations in mineral processing technology. Ore processing must be organized in such a way as to ensure the mineral liberation with optimal grinding of the ore. This process largely determines both the completeness of the extraction of minerals and elements from rocks, and the kinetics of various heterogeneous processes involving solid substances in a finely dispersed state.

Attempts to use the process of mechanical activation to increase the efficiency of mineral raw materials have been made for a long time. Specialized and generalizing works have shown that with an increase in dispersion leading to an increase in the substance activity, it is possible to intensify many technological processes: ore liberation, leaching of individual components from minerals, complete extraction of valuable elements from refractory ores and concentrates.

Mechanical activation, to some extent, leads to a change in the structural and physicochemical properties of minerals, especially surface ones. The research has ensured a fairly complete list of physicochemical changes in mineral matter and the transformation of materials during dispersion.

An analysis of works on the mechanochemistry of minerals shows the variety and complexity of the physicochemical processes during mechanical activation, and leads to the conclusion that the reactivity of minerals is determined both by a change in the fine crystal structure and an increase in the surface, and by solid-phase reactions, i.e. the processes accompanying mechanical processing.

Key words: gold-bearing raw materials, gold, flotation concentrate, grinding, mechanochemical processing, sedimentary test, leaching, sulfite-thiosulphate leaching, solution, cake, reduction-oxidation potential.

Introduction.

The technology of hydrometallurgical processing of gold-bearing sulfide raw materials includes two processingstages. At the first stage of the process, the raw material is oxidized andsulfur, iron and arsenic are converted into elements with a higher degree of oxidation. This is associated with the destruction of gold-retaining matrices of minerals (pyrite, arsenopyrite) and ensures more favorable conditions for the access of reagents to the particles of gold leached from the raw material. The success of the second stage of processing of raw materials, directly associated with the conversion of gold into solution, mainly depends on the oxidation rate of the raw material. If a sulfite-thiosulfate composition is used as a reagent for gold dissolving, this condition is slightly specific, different from other reagents. This is related to the fact that in this case sulfur is present as the main element in the rock-forming sulfidic minerals and reagent.Moreover, as was indicated in our previous publications [1-3], sulfur in combination with hydrothermal water are the “culprits” of gold deposits formation. Sulfur and water that inthe natural environment form various oxygen-free and, in the presence of air, oxygen chemical compounds (hydrosulfides, sulfides, thiosulfates, etc.), in some cases became gold solvents, in others, in combination with gold, transformed into an insoluble state. Thus, on its way from the subsoil to the earth’s surface, gold in compounds with sulfur, depending on the ph and Eh of the medium, was subjected to repeated dissolution and precipitation. These natural processes prove that it is possible to use sulfur, which is present in sulfide raw materials, partially or completely as a reagent in the technology of gold recovery in the processes of its dissolution and precipitation from solutions.

Materials and methods. In previous studies, the subject of our research was the autoclave oxidation processes of sulfide raw materials. The so-called redox process takes placeat high temperature (250-300ºC) and pressure (16-20 atmg), in a nitric acid medium in the presence of pure oxygen. The process requires high capital and operating costs. Gold is recovered from the solid residue by means of leaching using cyanide solutions.Sulfur in the autoclave is oxidized to a maximum 6-valent state and is completely disposed of in the form of ballast sulfate compounds. The analogous so-called nitrox or arsenic processes differ from the previous process in that they take place without pressure in ordinary conditions and in the atmospheric air. However, in this case, due to the lack of oxidizing potential, sulfide sulfur is oxidized only to an intermediate phase - the elemental state, which is the reason for low gold recovery atthe subsequent cyanidation of the solid residue, because elemental sulfur deposited on the surface of gold particles makes it difficult for the solvent to reach them. Within the frameworks of our works on this topic, dueto the use of an alkaline medium in the autoclave instead of an acidic one, it was possible to carry out this process in a milder mode (5 atmg instead of 20; 100-120ºC instead of 250-300). The negative influence of the partially elemental sulfur formed in this process was avoided due to the use of sodium sulfite with a small addition of thiosulfate as the reagent for leaching of the solid residue. The interaction of sodium sulfite with elemental sulfur results inthe formation of sodium thiosulfate, and thereby in the removal of the sulfur film from solid particles and simultaneous regeneration of the main reagent for gold leaching. Thus, in addition to solving an important technological problem, it was possible to makea part of the sulfur from the source sulfide raw material a useful element instead of the ballast product. In general, with approximately comparable parameters of gold recovery using the cyanide process, in this case, there is an additional effect of reducing the environmental load and economic costs.

Experimental. We continued working on the topic. The first stage of the autoclave oxidation process of sulfide raw materials was replaced with a simpler version of the mechanochemical processing carried out by means of finer grinding of the source sulfide material. Publications on the mechanochemical conditioning of sulfide raw materials before gold leaching are well systematized in [4]. The initial stage of pyrite oxidation is the formation of ferrous sulfate and elemental sulfur:

$$FeS_2 + 2O_2 = FeSO_4 + S \qquad (1)$$

At the same time, it is well known from popular manuals on general chemistry [5] that elemental sulfur easily interacts with alkalis and forms the whole range of oxygen and oxygen-free sulfur compounds:

$$3S + 6NaOH = 2Na_2S + Na_2SO_3 + 3H_2O \qquad (2)$$

$$S + Na_2SO_3 = Na_2S_2O_3 \qquad (3)$$

$$Na_2S + H_2O = NaHS + NaOH \qquad (4)$$

These reactions prove the possibility of formation of sodium thiosulfate and hydrosulfide compounds during the grinding of raw materials in an alkaline medium and the high probability of formation of water-soluble gold complexes with the mentioned sulfur anions, as evidenced by many publications. It is suffice to cite one of them [3], which seems to be the most interesting in connection with the fact that the mentioned processes are considered more extensively and are used by the authors as the evidence for their theory related to the formation of hydrothermal gold deposits using these processes.

According to this theory, during the formation of hydrothermal solutions at deep horizons in the negligible presence of oxygen and low ORP, gold was most likely present in solutions in the form of a hydrosulfide complex (AuS-). With the rise of solutions to the surface and the ORP increase, this complex was destroyed resulting in the separation of gold, which is commonly called early or high-temperature gold. At the same time, favorable conditions were created for the formation of the thiosulfate sulfur compound and the corresponding water-soluble thiosulfate gold complex. Subsequent more profound changes in the Eh-ph values of the medium lead to another destruction of this complex resulting in the formation of a gold deposit. The diagrams taken from the cited article and shown in Figures 1, 2, 3 are in good agreement with the author's conclusions and give a complete picture of the zones of stable existence of soluble gold compounds in the Eh-ph coordinates and the dependence of these compounds' activity in water on the ORP value. These data provide good guidance for planning and evaluating the results of current and future technological research on this topic.

For technological research, we used a 2.3-liter KazNITU laboratory ball mill. The research method involves grinding the concentrate in an alkaline medium in the presence of atmospheric oxygen as an oxidizer. Due to the absence of continuous air supply in the mill design, the mill must be stopped during the grinding process for periodical supply of fresh air through the loading chute.

The flotation concentrate for the study was obtained from Akbakaysky mining and processing plant. The concentrate has the following parameters: particle size: 0.074 mm, Au content: 15.5 g/t, As: 0.058%, S: 22.6%. Each 300 g of the initial sample of the concentrate loaded into the mill was added with 600 ml of caustic solution with a concentration of 143 g/L. Also, steel balls are loaded at a 1:10 ratio of the concentrate weight.The grinding was performedfor 60 minutes. Themill was stopped every 2 minutes for air supply. The measuredphvalues of the medium equaled 9.0-9.5. At that, the ORP varied within Ehvalues from -0.176 to -0.186V. Table 1 contains the results of sedimentation analysis of the source and ground products.

Table 1 – Sedimentation analysis

Values with regard to the source product (concentrate)		
µm	gram	%
+74	14.16	14.67
74-40	18.55	19.22
40-20	18.48	19.14
20-10	31.78	32.92
10-0	13.56	14.05
Total	96.53	100.0
Values of the sedimentation analysis after the grinding		
µm	gram	%
+74	21.58	25.63
74-40	1.2	1.43
40-20	14.18	16.88
20-10	17.95	21.37
10-0	29.1	34.63
Total	84.01	100.0

As follows from the Table, in the ground product there is a sharp increase in the 10 µm fraction (from 14.05 to 34.63%) and +74 µm fraction (from 14.67 to 25.63%).

These, at first glance, contradicting results have an adequate scientific explanation. According to the existing concepts [7, 8], mechanochemical activation of products is characterized by three stages of the process. While at the first stage there is practically no interaction between the ground particles, at the second stage the interaction begins to increment and is manifested in their aggregation due to Van der Waals forces. At the third stage, this process stops and gives way to the formation of chemical bonds between the particles. Based on these concepts, the +74 µm fraction, obtained as a result of grinding, was classified as the aggregated thinner class. So, it can be assumed that the overall increase in the content of the material with a fineness over 10 µm in the final product amounted to over 60%.

During the grinding in line with the above-mentioned mechanisms, gold was partially leached using reagents formed from its own sulfur. As a result, the mass content of gold in the final ground product significantly decreased from 15.5 g/t to 13 g/t, which corresponds to about 16% gold recovery rate into the solution right at this stage.

It is well known that adding cupric ions to the process of gold dissolution accelerates the process due to gold oxidation. Therefore, an experiment was further conducted with the addition of copper sulfate in the grinding process of sulfide concentrate (at a rate of 1 g/Lin terms of copper), while maintaining all the previous parameters of grinding. It resulted in the decrease in the gold content in the ground product to 8.3 g/t, i.e. the gold recovery rate into the solution at the grinding stage increased from 16 to almost 48%. A 100 g sample weight of the obtained ground product after separation of the liquid phase was subjected to sulfite-thiosulfate leaching in a 2-liter magnetic mixer in the following mode:

-Solid to liquid ratio is 1:10;
- Sodium sulfite content in the solution is 100 g/L;
- Sodium thiosulfate content in the solution is 50 g/L;
- Copper content in the solution is 1g/L;
- pHof the medium is 9-9.5,Eh varies from +0.034 to+0.084V;
- Leaching time is 4 hours.

For chemical analysis of theobtained leachate, a 240FS AA Agilent atomic absorption spectrometer was used, a C933 ionomer was used for measuring ph and Eh.

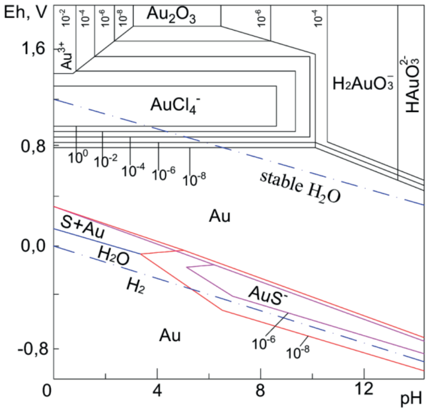

Figure1 - Stability ratios between some gold compounds in water at 25°C and 1 atm.

The amount of dissolved chloride components is 10^{-1} mol/L, and the amount of dissolved sulfur is 10^{-1} mol/L (according to Garrels).

Cake tests after leaching showed that its gold content was 6.1 g/t, i.e. additional gold recovery rate into the solution at this stage was about 27%.

Parameters of gold recovery into the solution with such simple instrumentation could be considered acceptable, however some negative factors associated with this method diminish the advantages of the technology. The ph and Eh measurements during the experiments showed that gold leaching is mainly done in line with the hydrosulfide option, and the zone of stable existence of the soluble hydrosulfide complex of gold in our case is approximately in zone “A” in Figure 2.

As Figure 2 shows, the range of gold existence in such a compound is characterized by a relatively narrow range of Eh values of the medium. It is difficult to maintain the parameters of real technological processes in this range. And taking into account that these processes take place mainly on the surface of the earth, the situation is complicated by the inevitable contact of productive solutions with atmospheric oxygen, which, in turn, can lead to uncontrolled deposition and loss of gold between the treatment stages.

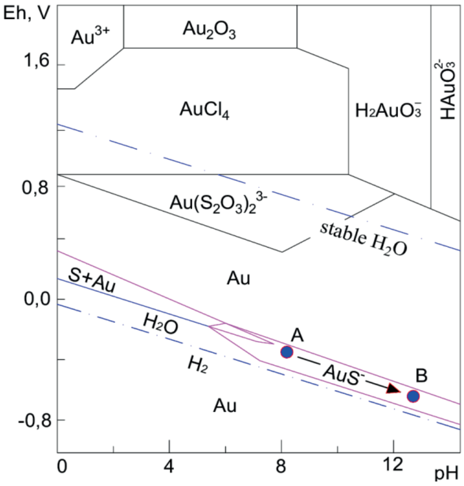

Figure2 -Stability ratios between some gold compounds in water at 25°C and 1 atm with due account for the equilibrium activity of thiosulfate ions.The amount of dissolved sulfur is 10^{-1} mol/L and the amount of dissolved chloride components is 10^{-1} mol/L.

Indeed, the calculations done by the authors of work [3] and presented graphically in Figure 3 show that the range of existence of water-soluble hydrosulfide gold complex is limited by narrow Eh values from -0.470 to -0.170V, which especially stand in contrast with the parameters of the thiosulfate compound, where such a zone is much wider and ranges from -0.170 to + 0.760V.

Apprehensions regarding the foregoing risks at the implementation of the hydrosulfide method of gold recovery were confirmed as a result of another experiment. With the purpose of increasing the dissolution of sulfide and elemental sulfur according to reactions 1–3, the experiment was performed with a higher dosage of alkali (ph above 11). It was expected that the process of formation of the hydrosulfide gold complex will continue more intensively and proceed in the direction indicated in Figure 2withthe arrow towards point “B”. However, the result turned out to be negative with a significant decrease in the gold dissolution. Most likely, the ORP parameters of the medium went beyond the range of existence of the soluble hydrosulfide gold complex. Another reason for the failure could also be a sharp reduction in the formation of a complexing hydrosulfide anion as a result of the reversal of the sodium sulfide hydrolysis [8] due to the creation of a considerable excess of alkali in the solution.

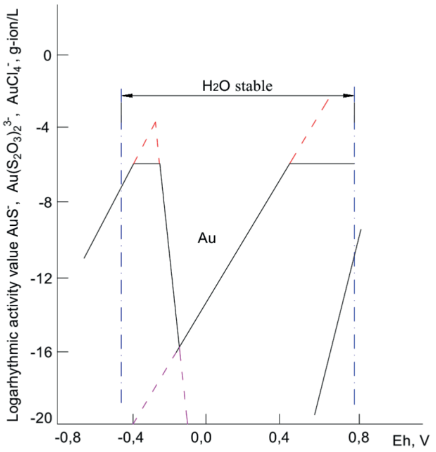

Figure3 -Dependence of the activity (concentration) of some gold compounds in water on the value of the oxidation-reduction potential at 25°C and 1 atm., ph = 8.0. The amount of dissolved sulfur is 10^{-1} mol/L.

Results and discussion.

Obviously, the practical creation of more favorable conditions for technological processes requires a transition to another zone - the zone of stable formation of the thiosulfate gold complex. According to calculations, this area in Figure 3 is located at ph = 8, for example, in a wider Eh range from -0.170 to +0.760. True, transiting into this area with the use of atmospheric oxygen as an oxidizer requires pressure, and in normal conditions even stronger oxidizer will be required. For this purpose, for example, chlorine can be used as a reagent, which in its active and ionic forms can act bothas an effective oxidizing and complexing agent in a water-soluble gold compound (Figure 1). In addition, the chlorine production in anactive sodium chloride water solution using the electrochemical method is a relatively simple task.

Conclusion. The mechanochemical activation by means of grinding a factory sulfide gold-bearing concentrate in the alkaline medium has been studied. The process was done using the 2.3-liter laboratory mill at 1:2 solid to liquid ratio and steel balls at the ratio of 1:10 to the concentrate weight. During 30 minutes of the grinding time, the -10 µm class solid material increased from 14 to 60%. At the measured parameters of the ground medium (ph=8–9 and Eh varies from –0.18 to –0.19V), approximately 16% of gold was recovered into the liquid phase of the mill. With the addition of up to 1 g/L of cupric ions to the grinding process, the recovery into the liquid phase increased up to 48%.

It was established that the conversion of gold into solution during the grinding process is explained by the formation of a water-soluble hydrosulfide gold complex $(AuS)^-$.

The remainder of the solid ground product was leached with a sulfite-thiosulfate reagent at 1:10 solid to liquid ratio, the content of sodium sulfite was 100g/L, sodium thiosulfate 50 g/L, copper 1 g/L, at ph = 9-9.5 and process time was 4 hours. A continuous supply of air into the reaction medium ensured maintaining the Eh value of the medium during leaching at a level of +0.030 to + 0.080V. As a result, about 27% of gold was additionally recovered into the solution.

The results of the performed study were deeply analyzed. It is shown that the practical application of the obtained data in the technology of gold recovery is associated with certain risks due to the relatively narrow range of sustainable existence in the conditions of mechanochemical activation of the raw materials of the hydrosulfide gold complex.

Further work will be directed at the optimization of processes at both stages of the developed technology. First of all, at the stage of mechanochemical activation of raw materials the work will be aimed at the increase in the oxidizing potential of the medium for converting gold in solution into a more stable compound. To this end, various methods of influencing the reaction medium, including electrochemical ones, will be tested.

Бегалинов Ә., Шәутенов М.Р., Медеуов Ч.Қ., Әлменов Т.М., Бектұр Б.Қ.

Сәтпаев университеті, Алматы, Қазақстан.
E-mail: bekturbek@bk.ru

ҚҰРАМЫНДА АЛТЫНЫ БАР СУЛЬФИДТІ ШИКІЗАТТЫ ӨҢДЕУ ПРОЦЕСІН МЕХАНОХИМИЯЛЫҚ БЕЛСЕНДІРУ

Аннотация. Ұсақтау процесіне мыс сульфатын қоса отырып, құрамында алтын бар флотациялық концентратты механохимиялық активтендіру бойынша зерттеулер жүргізілді. Механоактивация нәтижесінде бастапқы шикізатта өзіндік күкірт есебінен алтынның ішінара шаймалануы жүреді, ол шаймалаушы реагент рөлін атқарады. Ұнтақтау сатысында алтынды ерітіндіге айналдыру 48% құрады.

Өңдеу – пайдалы қазбаларды өңдеу технологиясындағы маңызды операциялардың бірі. Кенді дайындау –кенді оңтайлы ұсақтау кезінде минералдардың ашылуын қамтамасыз ететіндей етіп ұйымдастырылуы тиіс. Бұл процесс негізінен тау жыныстарынан минералдармен элементтерді алудың толықтығын, сондай-ақ қатты заттардың қатысуымен жұқа дисперстік үйде болатын әртүрлі гетерогенді процестердің кинетикасын анықтайды.

Минералды шикізаттың тиімділігін арттыру үшін механикалық активтендіру процесін қолдану әрекеттері бұрыннан белгілі. Арнайы және жалпылама жұмыстар заттың белсенділігінің артуына әкелетін дисперсияның жоғарылауымен көптеген технологиялық процестерді күшейтуге болатындығын көрсетеді: кенді ашу, минералды шикізаттан жекелеген компоненттерді сілтілендіру, тұрақты кендермен концентраттардан құнды элементтерді алу т.б.

Түйінді сөздер: құрамында алтыны бар шикізат, алтын, флотациялық концентрат, ұсақтау, механохимиялық өңдеу, седиментациялық талдау, шаймалау, сульфит-тиосульфатты шаймалау, ерітінді, тотығу-тотықсыздану әлеуеті.

Бегалинов А., Шаутенов М.Р., Медеуов Ч.К., Алменов Т.М., Бектур Б.К.[*]

Университет Сатпаева, Алматы, Казахстан.
E-mail: bekturbek@bk.ru

МЕХАНОХИМИЧЕСКОЕ АКТИВИРОВАНИЕ ПРОЦЕССА ПЕРЕРАБОТКИ ЗОЛОТОСОДЕРЖАЩЕГО СУЛЬФИДНОГО СЫРЬЯ

Аннотация. Проведены исследования по механохимическому активированию флотационного золото содержащего концентрата с добавлением в процесс измельчения медного купороса. Установлено, что в результате механоактивации происходит частичное выщелачивание золота засчет собственной серы в исходном сырье, которая выполняет роль выщелачивающего реагента. На стадии измельчения извлечение золота в раствор составило 48%.

Механическая обработка относится к числу важнейших операций в технологии обработки полезных ископаемых. Рудоподготовка должна быть организованна таким образом, чтобы обеспечить раскрытие минералов при оптимальном измельчении руды. Этот процесс во многом определяет, как полноту извлечения минералов и элементов изгорных пород, так и кинетику различных гетерогенных процессов, протекающих с участием твердых веществ в тонкодисперсном состоянии.

Попытки использовать процесс механической активации для повышения эффективности минерального сырья, известны давно. Специальными и обобщающими работами показано, что с повышением дисперсности, приводящей к возрастанию активности вещества, представляется возможным интенсифицировать многие технологические процессы: вскрытие руд, выщелачивание отдельных компонентов изминерального сырья, до извлечение ценных элементов и зупорных руд и концентратов.

Механическое активирование в большей или меньшей степени приводит к изменению структурных и физико-химических свойств минералов, особенно поверхностных. Проведенные к настоящему времени исследования позволяют составить достаточно полный перечень физико-химических изменений минерального вещества и трансформации материалов при диспергировании.

Анализ работ по механохимии минералов показывает многогранность и сложность физико-химических процессов, возникающих при механической активации, и позволяет прийти к заключению о том, что степень реакционной способности минералов определяется как изменением тонкой кристаллической структуры и увеличением поверхности, так и процессами, сопровождающими механическую обработку – твердофазными реакциями.

Ключевые слова: золотосодержащее сырье, золото, флотационный концентрат, измельчение, механохимическая обработка, седиментационный анализ, выщелачивание, сульфит-тиосульфатное выщелачивания, раствор, кек, окислительно-восстановительный потенциал.

Information about authors:

Begalinov A. – Doctor of Technical Sciences, Professor of “Mining” department, Satbayev University, Almaty, Kazakhstan; E-mail: begalinov_a@mail.ru, https://orcid.org/0000-0002-4793-6207;

Shautenov M. – Candidate of Technical Sciences, Professor of “Metallurgy and Mineral Processing” department, Satbayev University, Almaty, Kazakhstan; E-mail: shautenov_m@mail.ru, https://orcid.org/0000-0002-0266-3882;

Medeuov Ch. – Chief Scientific Associate Professor of “Mining” department, Satbayev University, Almaty, Kazakhstan; E-mail: medeu38@mail.ru; https://orcid.org/0000-0003-2624-4524;

Almenov T. – Candidate of Technical Sciences, Associate Professor of “Mining” department, Satbayev University, Almaty, Kazakhstan; E-mail: almen_t@mail.ru, https://orcid.org/0000-0001-9674-8598;

Bektur B. – Master of Technical Sciences, Lecture of “Mining” department, Satbayev University, Almaty, Kazakhstan; E-mail: bekturbek@bk.ru, https://orcid.org/0000-0003-0510-4995.

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ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

Редакторы: *М.С. Ахметова, А. Ботанқызы, Д.С. Аленов, Р.Ж. Мрзабаева*
Верстка на компьютере *Г.Д.Жадыранова*

Подписано в печать 15.12.2021.
Формат 60x881/8. Бумага офсетная. Печать – ризограф.
4,6 п.л. Тираж 300. Заказ 6.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-19