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Development of the technology of integrated processing the Chelkar deposit potash ore

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ABSTRACT

With the huge explored reserves of potash salts in Kazakhstan, there is still no production of potash fertilizers, the demand for which is constantly growing. In this regard, research of processing of the largest Chelkar deposit ore into potash fertilizers and salts is an urgent problem. The article presents the research results of washed potash ore decomposition with nitric acid and nitric acid suspension filtration. The filtering properties and granulometric composition of the insoluble residue were studied, on what basis the decomposition mode with precipitate double washing was determined. To ensure good suspension filterability, washed, uncalcined ore should be used. Tests of a by-product, gypsum, as a gypsum binder were carried out, which confirmed its compliance with the normally hardening gypsum binder of the G-2 B grade. Advantage of the obtained gypsum is its environmental friendliness. Salts, which are chlorine-free water-soluble potassium-magnesium fertilizers have been obtained by crystallization from nitric acid solution. On the basis of results of experimental-and-laboratory tests, a basic flow scheme has been developed for obtaining potash and complex potassium-nitrogen-magnesium fertilizers from the Chelkar deposit ore.

Keywords: carnallite, Chelkar deposit, potash fertilizer, washing, decomposition, evaporation

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Introduction

Potassium is one of the most important nutrient elements for increasing crop yields. The potash fertilizer market has been steadily developing without significant declines for several decades. World consumption of potash fertilizers varies between 45-52 million tons of KCl or 28.4-32.9 million tons of K_2O. Although potassium chloride and potassium sulfate still hold the lion's share of the market, other potassium compounds also play an important role in crop production [[1], [2]]. Potassium sulfate is of the greatest importance as a chlorine-free fertilizer that combines well with micronutrients.

Most of developed potassium deposits are concentrated in Canada (Saskatchewan), in Russia (Verkhnekamskoye field), in Belarus (Starobinskoye field). Most common minerals in them are sylvine - 63%, sylvinite - 12-15%, carnallite - 17%, cainite - 19%, langbeinite - 23%, polyhalite - 16%. Rocks contain impurities of other salts such as gypsum, carbonates, clay particles. Of 120 potassium-containing minerals, only a small part is of industrial importance [[3], [4]].

Currently, Kazakhstan mineral fertilizer industry is at the stage of accelerated development and expanding the product range [5]. Despite the fact that there are huge reserves of potash ores in country's subsoil resources, there is no production of potash salts and fertilizers in the country. Poor knowledge of the ores' composition of known deposits and methods of their processing is the reason for this. The unique raw material base includes more than 6 billion tons of proven reserves of potassium chloride in the form of sylvinite, carnallite-sylvinite and polyhalite. Powerful deposits located in Aktobe, Atyrau and West Kazakhstan regions are considered as ones of the largest in the world. "Satimola", "Chelkar", "Inder" and "Zhilyansk" domes are the most studied ones [6]. Development of Zhilyansk and Chelkar potassium salts' deposits is the most ambitious of the projects proposed for implementation, for which "Kazakhstan Potash" received subsoil use rights in 2011. These deposits are reportedly owned by "Kazakhstan Potash" through a local subsidiary, "Batys Kaliy" [7]. Carnallite is a main component of the Chelkar deposit ore, and there are also 5-15% sylvite and 15-25% halite. The company carried out geological exploration and estimation of potash ore reserves. But until now, enterprises for potash salts' mining have not yet moved to the production stage. Considering the growing demand for potash in the country and in the world, mining and use of domestic potash ores are becoming increasingly important [6].

The authors [8] investigated the possibility of obtaining potassium sulfate from polyhalite ore of the Zhilyansk deposit by washing it with water from halite, calcining and dissolving in water, followed by gypsum separation. A review of scientific publications did not reveal information about the results of the study of Chelkar deposit potash ore by other researchers. The search for methods of Chelkar ore processing into potash fertilizers and salts is an urgent problem of potash fertilizer industry formation in Kazakhstan.

Preliminary study of the ore composition carried out by us earlier, showed that the natural salt has a complex non-uniform composition, it contains, in addition to the main potassium component, a large amount of halite, as well as glaserite and an insoluble residue in the form of calcium sulfate dihydrate [9]. The mineral halite is an impurity component in potash fertilizers, the removal of which from the ore composition will permit to enrich it in potassium [[10], [11], [12]]. Therefore a process of the ore washing from halite by the method of incomplete dissolution was studied and the optimal enrichment conditions were determined. In order to reach complete dissolution of potassium and magnesium salts, in particular, sulfates, the methods of dissolving a calcined ore with hot water and decomposition of washed raw materials with nitric acid were investigated. It has been established that the ore calcination although leads to the destruction of crystalline hydrates, but does not allow to complete transfer useful components into a soluble state for obtaining a water-soluble fertilizer. The operating parameters of nitric acid decomposition of the concentrated ore have been determined, namely temperature of 50°C, nitric acid concentration of 20%, the process time is 30 minutes, at which potassium and magnesium salts' complete dissolution takes place [9]. A formed suspension is separated by filtration into a nitric acid extract and an insoluble gypsum precipitate. To determine the conditions for obtaining well-filtering crystals of calcium sulfate dihydrate, an additional study of the process of washed ore nitric acid decomposition and formed suspension filtration was carried out.

Experimental part

Chelkar deposit potash ore is an object of the study. Nitric acid suspension obtained by decomposition of the ore washed from sodium salts was separated by filtration in a vacuum filtration plant under a vacuum of 0.06 MPa. The filter cake was washed with hot water; filtration productivity and precipitate washing performance were determined by dry washed precipitate. For the study, flame photometric and spectrophotometric methods of analysis were used. Microscopic spectral analysis of salts was carried out using a JSM-6490l V scanning electron microscope (Jeol, Japan). Semi-quantitative X-ray analysis of solid phase samples was carried out on a D8 Advance apparatus (Bruker). Processing of the obtained data of diffraction patterns and calculation of interplanar distances were carried out using the EVA software. The obtained solid waste was tested as a gypsum binder. To determine the setting time a Vic's OGTs-1 device was used, the compression strength was determined using a PGM-100MG4A press. Sedimentation analysis of the insoluble residue was performed using a FSK-6 photo sedimentometer. The total standard measurement uncertainty and standard deviation for liquid and solid composition analyzes is calculated from 3 replicate measurements of the sample, taking into account sample weighing, calibration, and measurements on the flame photometer and spectrometer.

Results and discussion

The results of experimental laboratory tests of processing the Chelkar deposit potash ore show that the use of pre-calcined washed ore for nitric acid decomposition leads to formation of poorly filterable, practically non-separable suspension. At that the filtration productivity is 160 kg/m^2·h by dry precipitate (Table 1, experiment 1), which is obviously associated with gypsum dehydration and it transition to finely dispersed anhydrite during ore calcination. X-ray phase analysis of the dried, washed insoluble residue identifies calcium sulfate mainly in the form of anhydrate (76%) with gypsum content of 21% (Figure 1) [9].

Table1 - Indicators of the filtration process of nitric acid extract

Experiment	Composition of insoluble residue, %			Filtration productivity, kg/m^2·h	Average square particle diameter, µm	Specific surface area, cm^2/g
	SO_4^{2-}	CaO	MgO			
1	66.05	35.60	0	160	31	4855
2	55.59	29.25	0.36	3200	70	702

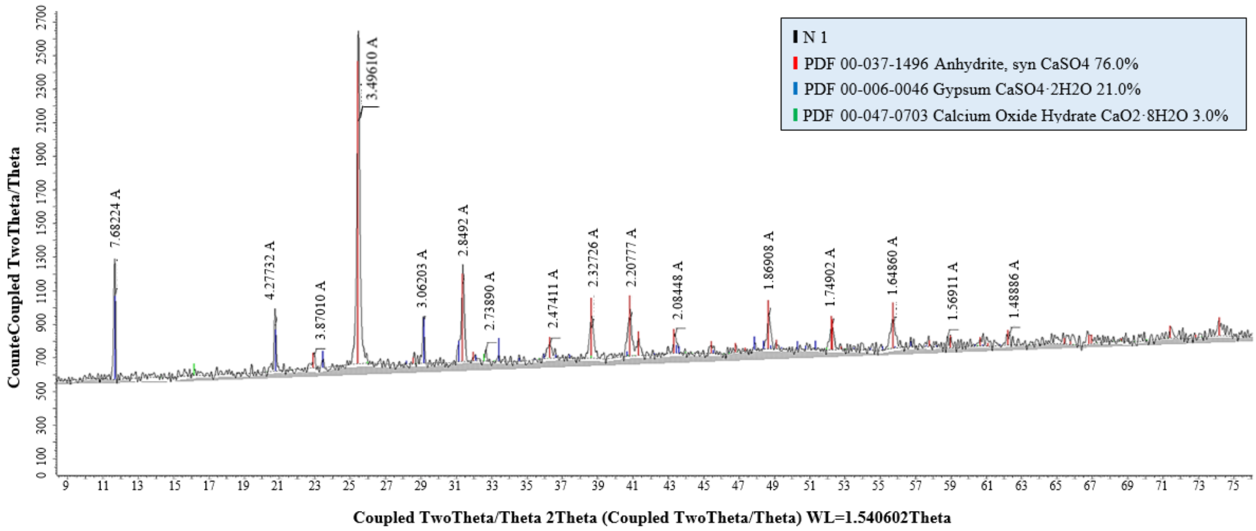

Figure 1 - X-ray diffraction pattern of the insoluble residue after nitric acid decomposition of calcined raw material

Fine-crystalline anhydrite clogs the filter pores and leads to significant deterioration in precipitate filtration properties [13]. To study possibility of eliminating this limiting stage and intensifying the filtration process, the decomposition of washed ore, which was not subjected to preliminary calcination, was investigated. At that as a result of nitric acid interaction, an insoluble gypsum precipitate remains in the solid phase, which is one of the components of natural potash ore; gypsum dehydration in this case does not occur. This leads to precipitation of coarse-crystalline precipitate and to increase extract filtration productivity by 20 times, up to 3200 kg/m^2·h by dry washed precipitate (Table 1, experiment 2). This suspension filterability increase is confirmed by sedimentation analysis of precipitates, which indicates an increase in particle size from 31 to 70 microns and a decrease in the specific surface of the precipitate by almost 7 times when using uncalcined ore instead of calcined one (Figures 2, 3; Table 1). As follows from Figure 2, about 50% of gypsum particles obtained in the first experiment have a root-mean-square diameter of 1-4 microns, while for the second experiment more than 30% of gypsum particles have a size from 60 to 100 microns. The difference in precipitate composition is also indicated by the results of spectral microscopic analysis (Figures 4, 5). Recalculation of composition of the main component calcium sulfate on the content of calcium and sulfur shows the presence of only calcium sulfate dihydrate (≈100% $CaSO_4·2H_2O$) in precipitate 2, and ≈90% of anhydrite ($CaSO_4$) in precipitate 1.

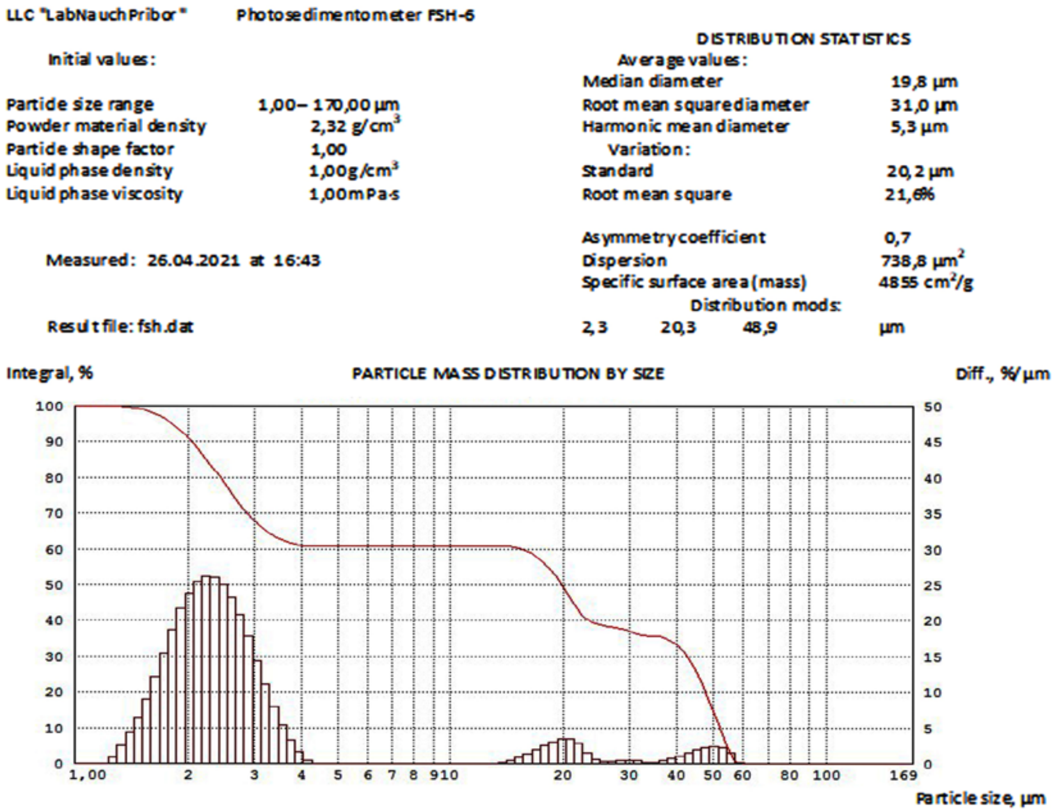

Figure 2 - Results of sedimentation analysis of calcium sulfate obtained by decomposition of calcined ore

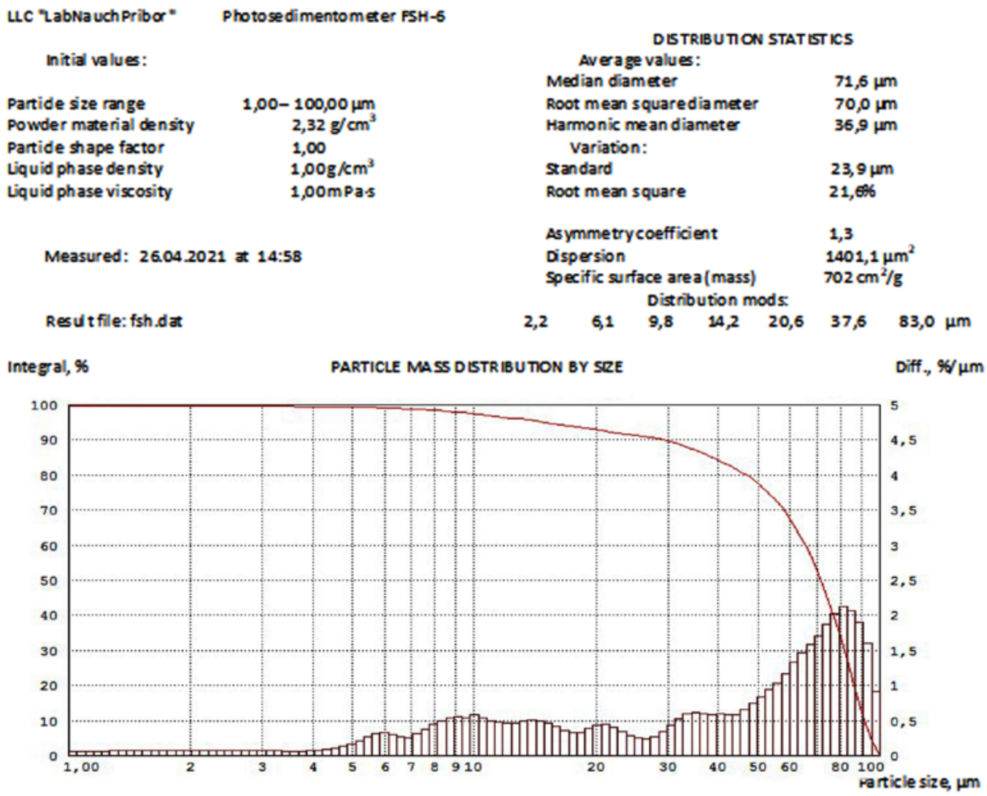

Figure 3 - Results of sedimentation analysis of calcium sulfate obtained by decomposition of uncalcined ore

Element	Weight,%
O	51.40
Al	0.18
Si	0.95
S	20.69
Ca	26.78
Total	100.00

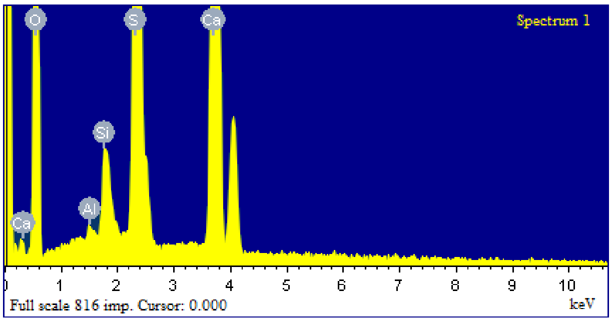

Figure 4 - Spectrogram of the insoluble residue obtained by decomposition of calcined ore (Sample 1)

Element	Weight, %
C	2.47
O	55.00
Mg	0.34
Si	0.74
S	18.04
Ca	23.41
Total	100.00

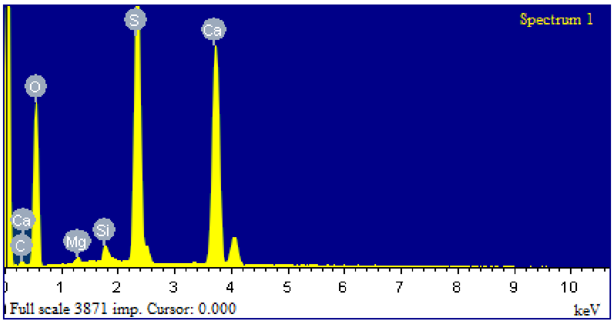

Figure 5 - Spectrogram of the insoluble residue obtained by decomposition of uncalcined ore (Sample 2)

The gypsum precipitate separated by filtration is a waste product, it does not practically contain impurities (Table 1) and therefore, after washing from mother liquor, it can be used in other chemical-technological processes. To wash out the insoluble residue, a counter-current two-stage washing scheme was used with water feed to the second stage and washing water to the first washing. The washing water obtained after the first stage of washing the insoluble residue was used for dilution to the required concentration of nitric acid fed for decomposition. The composition of filtrate and washing water is shown in Table 2.

Filtration productivity at the washing stages is at the level of this indicator for the main filtration. Crystallization of potassium-magnesium salts from nitric acid extract was carried out by partial solution evaporation at a constant temperature of 75°C and drying the separated crystals. At that the obtained crystalline product does not contain nitrogen, which remains in the mother liquor. The mother liquor was ammoniated to pH = 6 and dried to obtain nitrogen-potassium-magnesium chlorine-free water-soluble fertilizer in the form of sulfates and nitrates.

Table2 - Composition of the liquid phase after the decomposition of washed ore with 20% nitric acid

Liquid phase type	Content in liquid phase,%				Filtration productivity, kg/m^2·h
	K_2O	MgO	SO_4^{2-}	N	
Filtrate	4.53	2.54	10.91	2.03	3200
1 washing water	2.28	0.79	2.53	0.51	3320
2 washing water	1.21	0.36	1.71	0.20	3385

The dried gypsum by-product was tested to meet the standard requirements as low calcined gypsum binder. For this, the gypsum was calcined at a temperature of 160°C for 1 hour, then a gypsum dough of standard consistency was prepared and the setting time was determined on a Vic's device. For strength testing, samples of cubes were prepared, and 2 hours after the gypsum binder contact with water, the compression strength was determined using a press. Setting beginning of 8 minutes and setting end of 10 minutes were determined, as well as the compressive strength of the samples was 2.189 MPa. These indicators determine the tested binder as normally hardening gypsum binder (index B) of the G-2 B grade.

Experimental laboratory tests of the technology of studied potash ore processing confirmed the experimental results and allowed to establish optimal parameters for all stages of processing and to determine consumption coefficients for initial raw materials and reagents. Based on the results obtained, a basic flow scheme was developed for obtaining potash and complex potassium-nitrogen-magnesium fertilizers from the Chelkar deposit ore (Figure 6).

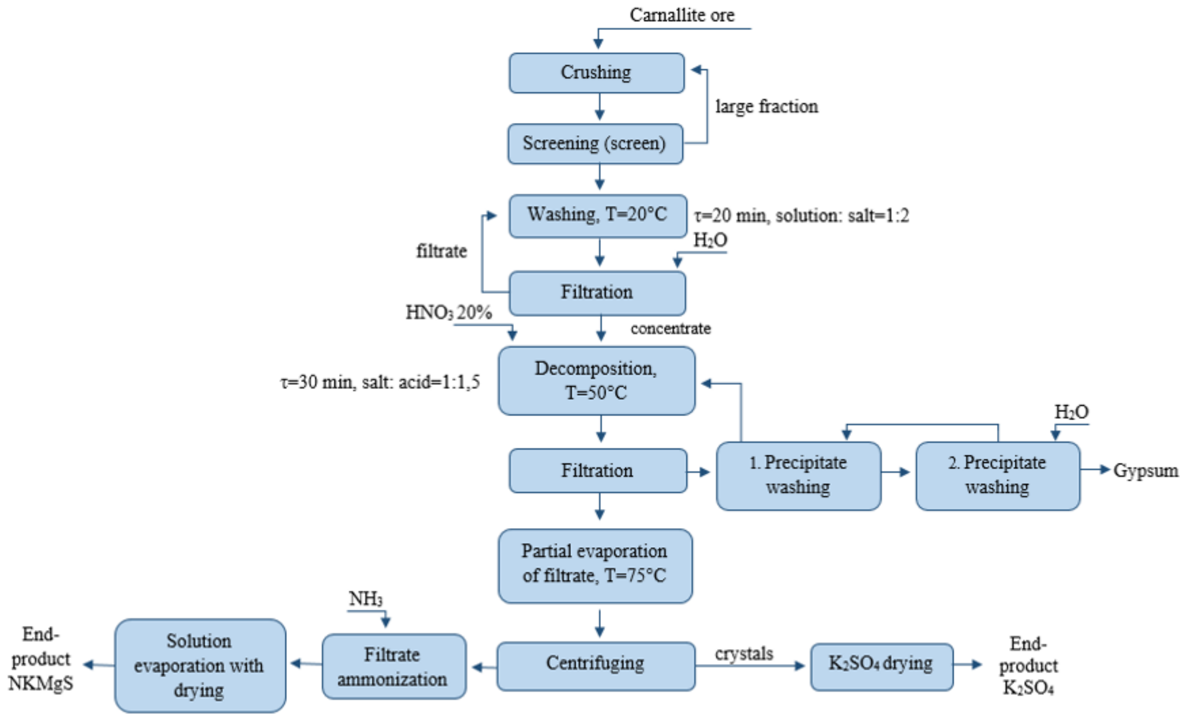

Figure 6 - Basic flow scheme forprocessing of Chelkar deposit potash ore

Crushed potash ore is washed from sodium salts in a screw dissolver with circulating mother liquor at a temperature of 20°C for 20 minutes with mass ratio of solution:salt = 1:2. The obtained suspension is separated in a settler, a clarified solution is returned to the dissolver for washing, and the thickened precipitate is fed to a vacuum filter and washed with water with the same L:S ratio. If necessary, a part of the solution can be evaporated to obtain a production salt, food sodium chloride. The washed ore is decomposed in an extractor with a stirrer with 20% nitric acid solution at a temperature of 50°C for 30 minutes with salt:acid ratio = 1:1.5. The suspension is separated by filtration on a vacuum filter. Washed with water and filtered cake, which is an insoluble gypsum residue, is sent for drying and further to the consumer. The washing water is returned to the reactor for nitric acid decomposition.The nitric acid extract is evaporated in an evaporator at a temperature of 75°C until crystallization from a solution of potassium and magnesium sulfate, which is then separated on a centrifuge. The mother liquor is ammoniated to pH = 6, evaporated and crystals are dried in a direct-flow drum dryer. The scheme is flexible and can be transformed to obtain only one type of nitrogen-potassium water-soluble fertilizer. The proposed technology is protected by an utility model patent "Method for processing potash ores to obtain potassium sulfate" [14].

Conclusions

As a result of completed research, the technology has been developed for obtaining potash and complex potassium-nitrogen-magnesium fertilizers from the Chelkar deposit ore. The stage of filtration of nitric acid suspension obtained by washed potassium ore decomposition with nitric acid has been studied. Filtration properties and particle size distribution of the insoluble precipitate were investigated, on the basis of which the decomposition mode was determined. Tests of dried gypsum showed its compliance with a normally hardening gypsum binder (index B) of grade G-2 B, which can be used as a binder in the construction industry for manufacture of gypsum plaster, partition wall plates and panels, decorative and other details in buildings and constructions. An advantage of the obtained gypsum is its environmental friendliness due to impurity absence in its composition. The obtained crystalline products do not contain soluble chlorides, they are completely water-soluble potassium-magnesium and nitrogen-potassium-magnesium fertilizers. An advantage of the developed technology is absence of solid and liquid production wastes and possibility to integrated use all components of the Chelkar deposit natural potash salt.

Conflict of interests

On behalf of all authors, the correspondent author declares that there is no conflict of interests.

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Челқар кен орнындағы калий кенін кешенді өңдеу технологиясын әзірлеу

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ТҮЙІНДЕМЕ

Қазақстанда калий тұздарының барланған орасан зор қоры бар болғанымен, үнемі сұранысы өсіп келе жатқан калий тыңайтқыштарын өндіру әлі де жоқ. Осыған байланысты ең ірі Челқар кен орнының кенін калий тыңайтқыштары мен тұздарына өңдеуді зерттеу өзекті мәселе болып табылады. Мақалада жуылған калий кенінің азот қышқылымен ыдырату және азот қышқылы суспензиясын сүзуді зерттеу нәтижелері берілген. Ерімейтін қалдықтың фильтрлеуші қасиеттері мен гранулометриялық құрамы зерттелді, соның негізінде тұнбаны қосарлы жуумен ыдырату режимі анықталды. Суспензияның жақсы сүзгіштігін қамтамасыз ету үшін жуылған, күйдірілмеген кенді пайдалану керек. Қосалқы өнім – гипстің гипс байланыстырғыш ретінде сынағы жүргізілді, бұл оның Г-2 Б маркасының қалыпты қататын гипс байланыстырғышына сәйкестігін растады. Алынған гипстің артықшылығы – оның экологиялық тазалығы. Азот қышқылы ерітіндісінен кристалдану нәтижесінде суда еритін хлорсыз калий-магний сульфатты тыңайтқыштарын көрсететін тұздар алынды. Тәжірибелік-зертханалық сынақтардың нәтижелері негізінде Челқар кен орнының кенінен калий және күрделі калий-азот-магний тыңайтқыштарын алудың үздіксіз технологиялық сызбасы әзірленді.

Түйін сөздер: карналлит, Челқар кен орны, калий тыңайтқышы, жуу, ыдырау, булану

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Разработка технологии комплексной переработки калийной руды месторождения Челкар

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АННОТАЦИЯ

При огромных разведанных запасах калийных солей в Казахстане до сих пор отсутствует производство калийных удобрений, спрос на которые непрерывно растет. В связи с этим исследование процесса переработки руды крупнейшего месторождения Челкар в калийные удобрения является актуальной проблемой. В статье приведены результаты исследования разложения отмытой калийной руды азотной кислотой и фильтрования азотнокислой суспензии. Изучены фильтрующие свойства и гранулометрический состав нерастворимого остатка, на основании чего определен режим разложения с двухкратной промывкой осадка. Для обеспечения хорошей фильтруемости суспензии следует использовать отмытую не прокалённую руду. Проведены испытания побочного продукта - гипса в качестве гипсового вяжущего, которые подтвердили соответствие его нормально твердеющему гипсовому вяжущему марки Г-2 Б. Преимуществом полученного гипса является его экологичность. Кристаллизацией из азотнокислого раствора получены соли, представляющие бесхлорные водорастворимые калийно-магниевые удобрения. На основании результатов опытно-лабораторных испытаний разработана принципиальная поточная схема получения калийных и сложных калийно-азотно-магниевых удобрений из руды месторождения Челкар.

Ключевые слова: карналлит, месторождение Челкар, калийное удобрение, отмывка, разложение, выпаривание

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