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ФИЗИКО-ХИМИЧЕСКИЕ И ТЕХНОЛОГИЧЕСКИЕ АСПЕКТЫ ПЕРЕРАБОТКИ КАОЛИНОВЫХ ГЛИН МЕСТОРОЖДЕНИЯ «ЧАШМАСАНГ» СПОСОБОМ СУЛЬФАТИЗАЦИИ

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В статье приведены результаты физико-химических исследований по сульфатизации каолиновых глин месторождения «Чашмасанг» с целью получения неочищенных и очищенных коагулянтов (сульфата алюминия), гидроксида алюминия и глинозема. Месторождение каолиновой глины «Чашмасанг» расположено на южном склоне Гиссарского хребта Таджикистана на водоразделе междуречья Хонака – Суффа и охватывает площадь 0,2 км^2. По ходу проведения исследований определены химический и минералогический составы каолиновых глин месторождения «Чашмасанг», содержание оксида алюминия, которое находится в приделах 19-22%, и в основном присутствует в виде минерала каолинита. Выявлены оптимальные технологические параметры, влияющие на процесс сульфатизации: температура 220-260 °С, продолжительность 60-90 мин, концентрация серной кислоты 90-95%, дозировка кислоты до 110% от стехиометрии Также был изучен процесс водной обработки сульфатизированного спека с целью получения очищенного смешанного жидкого коагулянта. Оптимальные параметры процесса следующие: температура 85-95 °С, продолжительность 40-45 мин, соотношение твердой и жидкой фаз (Т:Ж)=1:4. Максимальная степень извлечения сульфата алюминия в пересчете на глинозем составляет более 90%. Проведенным рентгенофазовым анализом подтверждено, что после сульфатизации каолиновых глин месторождения «Чашмасанг» появляются новые линии, которые характерны минералу миллозевичиту ($Al_2(SO_4)_3$) и алуногену ($Al_2(SO_4)_3 \cdot 18H_2O$). Результаты водной обработки сульфатизированного спека с целью получения очищенного сульфата алюминия были подтверждены рентгенофазовым анализам. Выявлено, что в составе твердого остатка после фильтрации пульпы присутствует оксид кремния в виде минерала кварца, и упаренная соль из жидкой части является безводным сульфатом алюминия (минерал миллозевичит).

Ключевые слова: каолиновая глина, каолинит, сульфатизация, сульфат алюминия, гидроксид алюминия, глинозем, коагулянт, серная кислота

PHYSICO-CHEMICAL AND TECHNOLOGICAL ASPECTS OF PROCESSING KAOLIN CLAYS OF CHASHMASANG DEPOSIT BY SULFATIZATION METHOD

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The article presents the results of physical and chemical studies on the sulfatization of kaolin clays of the Chashmasang deposit in order to obtain crude and purified coagulants (aluminum sulfate), aluminum hydroxide and alumina. The deposit of kaolin clay Chashmasang is located on the southern slope of the Gissar Range of Tajikistan on the watershed of the Khonaka-Suffa interfluve and covers an area of 0.2 km^2. In the course of the research, the chemical and mineralogical composition of kaolin clays of the Chashmasang deposit was determined, the content of aluminum oxide, which is in the aisles of 19-22%, and is mainly present in the form of the mineral kaolinite. The optimal technological parameters that affect the sulfatization process have been identified: temperatures 220-260 °C, duration 60-90 minutes, sulfuric acid concentration 90-95%, acid dosage up to 110% of stoichiometry. Also, the process of water treatment of sulfated sinter was studied in order to obtain a purified mixed liquid coagulant, the optimal parameters of which are as follows: temperature 85-95 °C, duration 40-45 min, ratio between solid and liquid (S:L) = 1:4. With such optimal parameters, the degree of extraction of aluminum sulfate in terms of alumina is more than 90%. The X-ray diffraction confirmed that after sulfatization of kaolin clays from the Chashmasang deposit, new lines appear that are characteristic of the mineral millosevicite ($Al_2(SO_4)_3$) and alunogen ($Al_2(SO_4)_3 \cdot 18H_2O$). The results of water treatment of sulfated sinter in order to obtain purified aluminum sulfate was confirmed by X-ray diffraction. It was revealed that silicon oxide in the form of quartz mineral remains in the composition of the solid residue after filtering the pulp, and the evaporated salt of the liquid part is anhydrous aluminum sulfate (millosevichite mineral).

Key words: kaolin clay, kaolinite, sulfatization, aluminum sulfate, aluminum hydroxide, alumina, coagulant, sulfuric acid

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INTRODUCTION

Kaolin clay is mainly used to obtain water purification coagulant (crude aluminum sulfate), $Al(OH)_3$, Al_2O_3, porcelain, faience, electrochemical and construction ceramics, refractory products, cement, etc. [1-6]. As for the high aluminium oxide content in kaolin clays, the Zhuravliny Log deposit (Chelyabinsky district, Russia) with reserves of more than 60 million tonnes and an aluminium oxide content of more than 36% [7], which is quite suitable for the production of the above-mentioned types of products, can be cited as an example. Based on the results of geological works and on temporary conditions, reserves of kaolin clays were calculated at the Chashmasang deposit (Tajikistan). The total geological reserves of kaolin clays in categories C, + C_2 are 1,958,892 t, including in category C – 478,190 t and C_2 – 1,480,702 t. Reserves of kaolin clays in the State Reserves Commission are not approved [8-9]. In works [10-12] were studied the physicochemical and technological bases for the processing of aluminum-containing ores – argillites of kaolin clays of the Chashmasang deposit in Tajikistan with various mineral acids, as well as the processes of decomposition of argillites by the chlorine method. In [13], the processing of nepheline syenites, kaolin clays, and argillites by sintering with calcium chloride and sodium hydroxide was studied.

One of the most effective ways of processing low-quality alumina-containing minerals is the acid method, in the process of acid treatment, silicon oxide remains in the insoluble solid part, while alumina and other useful components go into solution [14, 15]. Among the acidic methods of processing alumina-containing minerals, the use of sulfuric acid occupies a special place due to the relatively low effect on steel apparatus, the high degree of alumina yield and its relatively low cost [16-22]. The advantage of using sulfuric acid in the Republic of Tajikistan is that an enterprise for the production of sulfuric acid (LLC «TAlCo Chemical») operates here in the city of Yavan [23-24].

In the literature, the processing of kaolin clays from the Chashmasang deposit by the sulfatization method is absent. Therefore, the aim of the work is to study the physical and chemical parameters of the technology for processing kaolin clays from the Chashmasang deposit by the sulfatization method.

MATERIALS AND EXPERIMENTS

Based on this, the process of sulfatization of kaolin clay from the Chashmasang deposit was studied in order to obtain purified and unpurified coagulants, cryolite, aluminum hydroxide and alumina in laboratory conditions.

RESULTS AND ITS DISCUSSION

First of all, the chemical composition of kaolin clay from the Chashmasang deposit was studied, the results of which are given in Table.

Table

Chemical composition of kaolin clays from the Chashmasang deposit

Таблица. **Химический состав каолиновых глин месторождения «Чашмасанг»**

The content of the components, wt. %							
SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO	MgO	Na_2O	Loss on ignition
62.1	20.5	6.9	0.5	1.8	0.8	0.6	6.1

In order to confirm the chemical analyzes carried out, as well as to determine the mineralogical composition of the kaolin clay of the Chashmasang deposit, X-ray diffraction (XRD) was carried out on a modern DRON-2 device. It was revealed that the main lines belong to the mineral's kaolinite ($Al_2Si_2O_5(OH)_4$) (N 29-1488 from PDF card index file), quartz (SiO_2) (N 78-1254 from PDF card index file), and magnetite ($Fe_2O_3 \cdot FeO$) (N 72-1673 from PDF card index file).

After studying the chemical and mineralogical composition on the basis of stoichiometric calculations, the process of sulfatization of kaolin clay from the Chashmasang deposit was studied. When sulfatization is shown kaolin clay, the following chemical reactions are possible.

$$Al_2Si_2O_5(OH)_4 + 3H_2SO_4 \rightarrow \rightarrow Al_2(SO_4)_3 + 2SiO_2 + 5H_2O \quad (1)$$

$$Fe_2O_3 \cdot FeO + 4H_2SO_4 \rightarrow \rightarrow Fe_2(SO_4)_3 + Fe(SO_4) + 4H_2O \quad (2)$$

$$CaCO_3 + H_2SO_4 \rightarrow CaSO_4 + H_2O + CO_2 \quad (3)$$

$$MgCO_3 + H_2SO_4 \rightarrow MgSO_4 + H_2O + CO_2 \quad (4)$$

$$Na_2O + H_2SO_4 \rightarrow Na_2SO_4 + H_2O \quad (5)$$

$$K_2O + H_2SO_4 \rightarrow K_2SO_4 + H_2O \quad (6)$$

The main factors influencing the sulfatization process are: temperature, process duration, concentration and dosage of sulfuric acid. The research results are shown in Fig. 1 and 2 in the form of graphs. The influence of the temperature of the sulfatization process on the degree of extraction of aluminum sulfate (in terms of alumina) is shown in Fig. 1 (curve 1).

As can be seen from the Figure, with an increase in temperature, the rate of sulfatization of kaolin clay increases, and the degree of extraction of Al_2O_3 changes from 25.8 to 91.7% in the temperature range from 100 to 260 °C. However, an increase in temperature above 280 °C adversely affects the degree of extraction of alumina, which is associated with the evaporation and decomposition of sulfuric acid. On Fig. 1 (curve 2) shows the dependence of the degree of extraction of aluminum sulfate on the duration of the sulfatization process.

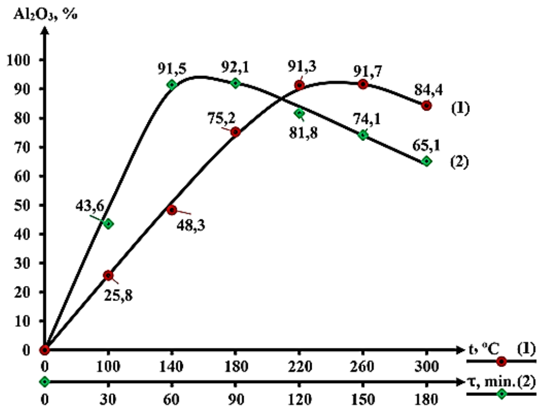

Fig. 1. The dependence of the degree of extraction of alumina on temperature (1) and the duration of the sulfatization process (2) at a constant concentration of sulphuric acid – 95% and its dosage – 100% of the stoichiometry

Рис. 1. Зависимость степени извлечения глинозема от температуры (1) и продолжительности процесса сульфатизации (2) при постоянной концентрации серной кислоты – 95% и ее дозировке – 100% от стехиометрии

For 30 min of the process, the degree of extraction of Al_2O_3 is 43.6%, and with an increase in time to 60-90 min, the degree of extraction of alumina steadily increases to 92.1%. With an increase in the duration of the process from 90 to 180 min, the degree of release of aluminum sulfate decreases from 92.1 to 65.1%, which is due to the decomposition of the formed aluminum sulfate with the formation of water-insoluble aluminum hydroxide and sulfuric anhydride in the form of gas. The temperature and duration of the process were varied at a sulfuric acid concentration of 95% and an acid dosage of 100% of the stoichiometry.

The influence of the concentration and dosage of sulfuric acid on the degree of extraction of alumina is shown in Fig. 2.

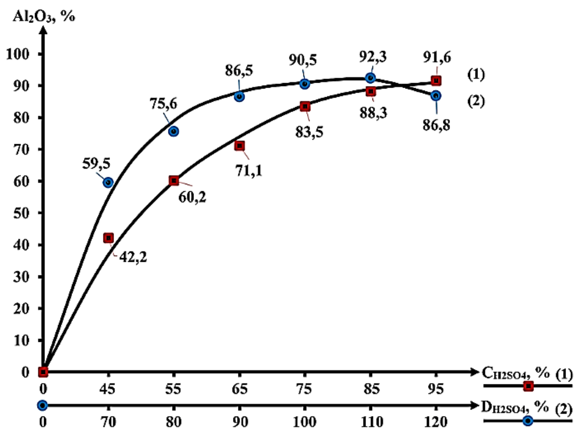

Fig. 2. Dependence of the degree of extraction of alumina on the concentration (1) and the dosage of sulfuric acid (2) at a constant process temperature of 260 °C and a duration of 90 min

Рис. 2. Зависимость степени извлечения глинозема от концентрации (1) и дозировки серной кислоты (2) при постоянной температуре процесса – 260 °C и его продолжительности – 90 мин

As can be seen from Fig. 2 (curve 1), when the concentration of sulfuric acid changes from 45 to 95%, the degree of alumina extraction increases from 42.2 to 91.6%. The effect of the dosage of sulfuric acid on the degree of extraction of alumina was determined on the basis of a stoichiometric calculation of the interaction of the components of kaolin clay with sulfuric acid, the result of which is shown in Fig. 2 (curve 2). It can be seen from the graph that when the dosage of sulfuric acid is changed from 70 to 110% according to the stoichiometric calculation, the degree of alumina extraction increases from 59.5 to 92.3%, and its additional increase leads to a decrease in the degree of alumina extraction. In our opinion, with an increase in the dosage of sulfuric acid by more than 110%, the viscosity of the mixture increases, the diffusion of sulfuric acid molecules decreases and, as a result, the interaction of acid molecules with kaolin clay molecules slows down, which in turn leads to a decrease in the degree of extraction of alumina.

As a result of the experiments, the optimal parameters for the process of sulfatization of kaolin clay from the Chashmasang deposit were established: temperature – 220-260 °C; the duration of the process is 60-90 min; H_2SO_4 concentration – 95-98%; dosage of sulfuric acid – 100-110% of the stoichiometric amount. Under such conditions, the degree of extraction of alumina is more than 90%.

In order to confirm the process of sulfatization of kaolin clay from the Chashmasang deposit, XRD of sulfatized sinter was carried out, the result of which is shown in Fig. 3.

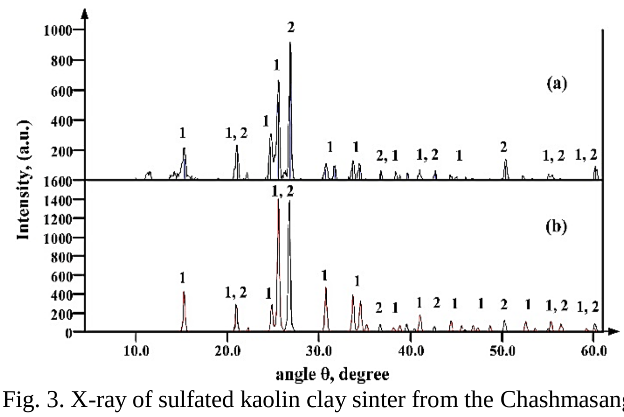

Fig. 3. X-ray of sulfated kaolin clay sinter from the Chashmasang deposit (a) and standard sample (б): 1-millosevicite ($Al_2(SO_4)_3$), 2-quartz (SiO_2)

Рис. 3. Рентгенограмма сульфатизированного спека каолиновой глины месторождении «Чашмасанг» (а) и стандартного образца (б): 1-миллозевичит ($Al_2(SO_4)_3$), 2-кварц (SiO_2)

XRD of the sulfated sinter (Fig. 3) shows that after the sulfatization of kaolin clay from the Chashmasang deposit, new lines appear that belong to the mineral millosevicite ($Al_2(SO_4)_3$) (N 22-21 from PDF card index file) and quartz (SiO_2) (N 78-1254 from PDF card index file). Since the degree of extraction of ferrous sulfates under optimal conditions is up to 40%, the lines belonging to ferrous sulfate are not observed on the X-ray pattern. Thus, the XRD confirms the process of sulfatization of kaolin clay from the Chashmasang deposit and proves the formation of aluminum sulfate. The resulting sulfated sinter can be used as a crude coagulant.

In order to separate soluble sulfates from sulfated sinter, the water treatment process was studied. The main parameters influencing the water treatment process are: temperature, process duration and the ratio of solid and liquid phases.

It was determined that when the temperature rises from 45 to 95 °C, the degree of aluminum sulfate release (in terms of aluminum oxide) increases from 41.2 to 92.1%, and with a change in the duration of the process from 20 to 40-50 min the degree of separation of alumina varies from 53.6 to 92.3%. With an increase in the duration of the process for more than 50 min, the degree of isolation of aluminum sulfate decreases, which is associated with the evaporation of the aluminum sulfate solution and its crystallization at a temperature of 95 °C.

At the same time, at a ratio of solid and liquid phases equal to S:L – 1:4-5, the degree of separation of aluminum sulfate is more than 90%, adding more water leads to a decrease in the degree of extraction of alumina, which is associated with hydrolysis of the resulting dilute solution of aluminum sulfate.

To confirm the process of water treatment of sulfated sinter after filtering the pulp, X-ray diffraction analysis of the evaporated sample of the aluminum sulfate solution and the solid residue was carried out. It was determined that the revealed X-ray lines of the evaporated solution belong to the mineral millosevicite ($Al_2(SO_4)_3$) and this confirms the formation of pure anhydrous aluminum sulfate from kaolin clay of the Chashmasang deposit. And also, the X-ray of the solid residue indicates that all the detected lines belong to the mineral quartz (SiO_2), and thus confirms the complete dissolution of the millosevicite mineral in water.

In order to establish the temperature changes of the obtained aluminum sulfate crystal hydrate ($Al_2(SO_4)_3 \cdot 18H_2O$), differential thermal analysis (DTA) was carried out on a LabSys Evo device at a speed of 10 °C/min, the result of which is shown in Fig. 4.

According to DTA (Fig. 4), the thermal decomposition of aluminum sulfate crystal hydrate proceeds schematically as follows.

$$Al_2(SO_4)_3 \cdot 18H_2O \xrightarrow{50-115℃}$$
$$\xrightarrow{50-115℃} Al_2(SO_4)_3 \cdot 8{,}5H_2O \xrightarrow{115-215℃}$$
$$\xrightarrow{115-215℃} Al_2(SO_4)_3 \cdot 4{,}5H_2O \xrightarrow{215-315℃}$$
$$\xrightarrow{215-315℃} Al_2(SO_4)_3 \cdot 1{,}5H_2O \xrightarrow{315-670℃}$$
$$\xrightarrow{315-670℃} Al_2(SO_4)_3 \cdot 0{,}5H_2O \xrightarrow{670-830℃}$$
$$\xrightarrow{670-830℃} \gamma - Al_2O_3 + 3SO_2\uparrow + 1{,}5O_2\uparrow + 0{,}5H_2O\uparrow$$

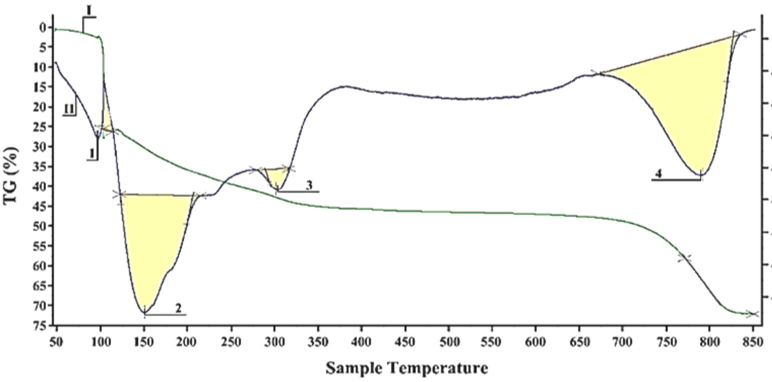

Fig. 4. DTA of aluminum sulfate crystal hydrate obtained by sulfatization of kaolin clay: I-TG line; II-DTA line; 1,2,3,4-endothermic effects

Рис. 4. ДТА кристаллогидрата сульфата алюминия, полученного сульфатизацией каолиновой глины: I-линия ТГ; II-линия ДТА; 1,2,3,4-эндотермические эффекты

As can be seen from Fig. 4, when the temperature changes from 25 to 830 °C, 4 deep endothermic effects appear on the DTA line (Fig. 4, II) (marks 1,2,3,4), characterized, respectively, by weight loss on the TG line (Fig. 4, I). According to the reaction scheme of the thermal decomposition of aluminum sulfate crystal hydrate and the TG/DTA line (Fig. 4, I, II), the first endothermic effect (Fig. 4, mark 1) occurs in the temperature range from 50 to 115 °C, which is associated with the loss of 9.5 water molecules of aluminum sulfate crystal hydrate and the transformation of $Al_2(SO_4)_3 \cdot 18H_2O$ into $Al_2(SO_4)_3 \cdot 8.5H_2O$. The second endothermic effect (Fig. 4, mark 2) occurs in the temperature range from 115 to 215 °C, which corresponds to the loss of 4.5 water molecules with the formation of $Al_2(SO_4)_3 \cdot 4H_2O$. With the third endothermic effect (Fig. 4, mark 3) in the temperature range from 215 to 315 °C, 3 water molecules are removed with the transformation of $Al_2(SO_4)_3 \cdot 4.5H_2O$ into $Al_2(SO_4)_3 \cdot 1.5H_2O$. Further heating in the temperature range from 315 to 670 °C leads to the loss of up to 1.0 water molecules and, accordingly, the formation of $Al_2(SO_4)_3 \cdot 0.5H_2O$. Thus, up to a temperature of 670 °C, the total water loss is more than 97%. According to the fourth deep endothermic effect (Fig. 4, mark 4), starting from a temperature of 670 °C, complete removal of molecular water ($0.5H_2O$) occurs and up to a temperature of 830 °C, the process of decomposition of aluminum sulfate is observed with the formation of gamma modification of alumina ($\gamma - Al_2O_3$), sulfur dioxide (SO_2), oxygen (O_2) and water vapor (H_2O), which, according to the TG line (Fig. 4, I), is associated with a 37% weight loss. As a result of the DTA studies, it turned out that during the heat treatment of aluminum sulfate crystal hydrate from sulfatized kaolin clays of the Chashmasang deposit to a temperature of 830 °C, technical alumina can be obtained.

CONCLUSIONS

The studies carried out make it possible to develop a technology for the extraction of aluminum sulfate (purified coagulant) from local alumina-containing raw materials, moreover, during the processing of aluminum sulfate, cryolite, aluminum hydroxide, alumina, which is mainly used in the production of metallic aluminum and fluorine-containing compounds.

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The authors declare the absence a conflict of interest warranting disclosure in this article.

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